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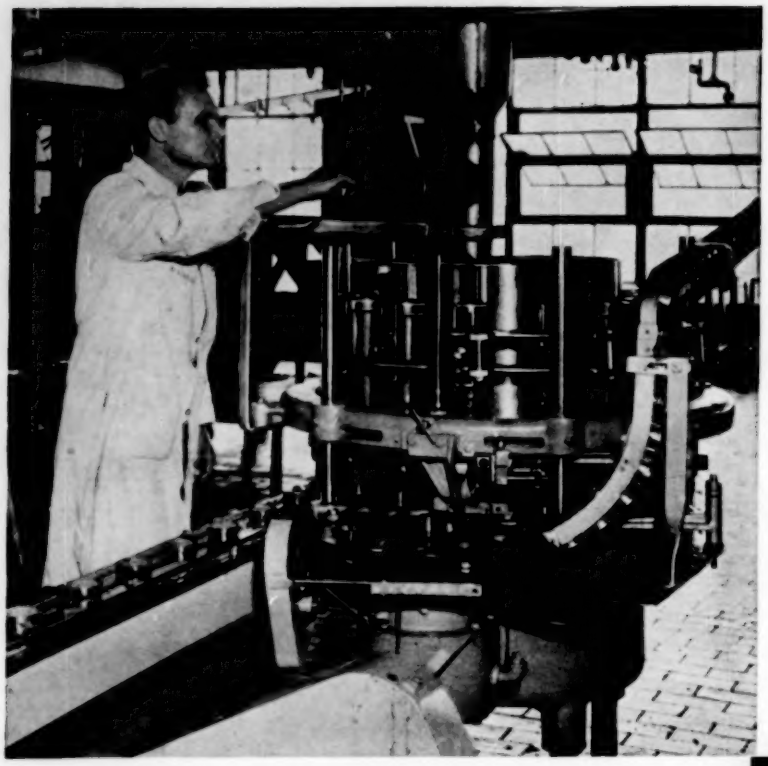

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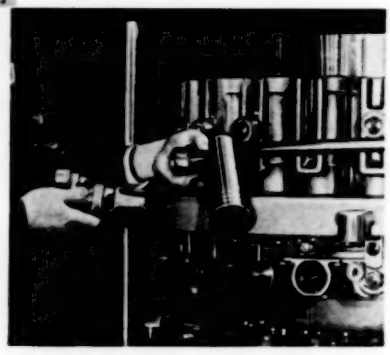

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Rapid Method for Moisture in Fruits and Vegetables by Oxidation With Dichromate. II. Pineapple-Rice Pudding, Rice, Prunes, and Corn

YOSHIO TOMIMATSU AND HERBERT F. LAUNER
Western Regional Research Laboratory,[a] Albany, California
(Manuscript received May 26, 1952)

The rapid, simple method for moisture determination in fruits and vegetables, based upon oxidation with potassium dichromate solution, has been applied to pineapple-rice pudding, rice, prunes, and fresh frozen corn. Data are presented on precision and accuracy.

In a previous paper *(2)* the writers reported that a simple method, based upon oxidation with potassium dichromate solution and requiring only 5 to 10 minutes per weighed specimen, can be used to determine the moisture content of potatoes and peas. Application to a considerable number of varieties of dehydrated potatoes and fresh, frozen peas resulted in mean errors of 0.37 and 0.43% moisture. Moisture values were shown to be in terms of the particular reference method used. The method was found to have optimum accuracy for materials of high moisture content.

In the present paper are presented results of applying the method to the determination of moisture in the following products with the approximate moisture percentages as indicated: pineapple-rice pudding (an Army ration item), 68; rice, 12 to 62; prunes, 17 to 68; and corn, 75.

ANALYTICAL PROCEDURE

Sample preparation. Test specimens for both the dichromate oxidation and reference moisture determinations were taken from a prepared representative sample. The corn and the dry rice were ground in an intermediate-size Wiley mill having a 20-mesh screen. Mixing the ground material with a spatula was sufficient to give reproducible results. The pitted prunes and the moist rice were mechanically blended with water to give a slurry of maximum smoothness and minimum separation during weighing. The moisture contents of prunes and moist, precooked rice listed in the tables refer to the moisture content before blending. The pineapple-rice pudding was mechanically slurried without added water.

Oxidimetric procedure. All samples of commodities except prunes[b] were analyzed in quadruplicate as follows:[c] To the specimen, 0.25 ± 0.02 g., dry basis, in a 400 ml. beaker, 25.00 ml. of 1.835 *N*. standard dichromate solution was added. Then 10 ml. of concentrated sulfuric acid, reagent grade, density 1.84, was slowly (10 to 15 seconds) added to the mixture covered by a watch glass; efficient stirring (preferably magnetic) was important. After the foaming had moderated, usually after 30 seconds, 30 ml. more of the acid was added rapidly, the mixture allowed to stand for 4 minutes, diluted with about 150 ml. of water, and the excess dichromate titrated electrometrically with ferrous ammonium sulfate solution *(2)*.

A standing time of 4 minutes (before stopping the oxidation by diluting with water) was found applicable to the materials studied because, in this time, the oxidations reach 99% or more of the final value. This is shown in Figure 1, in which the

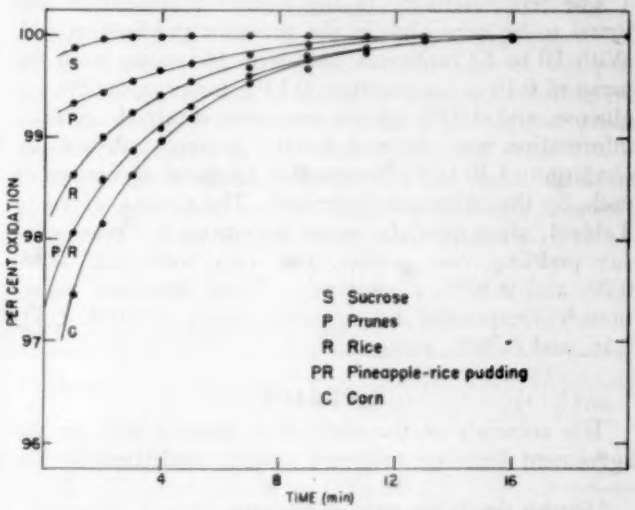

Figure 1. Rate of oxidation of various materials by dichromate under analytical conditions. All values are relative. The 5-minute, and subsequent, values for sucrose corresponded to 99.2% of theory (2).

curves for the various materials including sucrose for comparison, reach a constant value in 20 minutes or less, which furthermore was not exceeded during overnight standing. The points were obtained from aliquots of larger volumes of reaction mixtures.

It is apparent that a shorter standing time can be used if the variations therein are kept compatible with the accuracy desired, and if the same procedure is used for analysis and determination of the oxidation factor.

Reference moisture methods. A vacuum oven at less than 5 mm. Hg was used for all dry-weight determinations, which were run in triplicate. Pineapple-rice pudding was pre-dried for 2 hours at 100° C. in a forced-draft oven and then dried an additional 18 hours in a vacuum oven at 100° C. Dry rice was dried for 5 hours in a vacuum oven at 100° C. while moist precooked rice was pre-dried for 2 hours in a forced-draft oven and then dried an additional 25 hours in a vacuum oven at 100° C. Prunes were pre-dried for 2 hours in a forced-draft oven at 60° C., and then dried for 40 hours in a vacuum oven at 60° C. For prunes of normal moisture content (20-26% moisture), this method gave results comparable to the A.O.A.C. method *(1)*. However, for very dry prunes (11-14% moisture), the A.O.A.C. method gave moisture values which were several percent lower than those obtained by the reference method, probably because the prunes were so dry that subsequent mixing of the ground specimen with asbestos, as called for in the A.O.A.C. sample-preparation procedure, was very difficult despite the use of large amounts of water. Corn was pre-dried for 2 hours in a forced-draft oven and then dried for 40 hours in a vacuum oven at 70° C.

[a] Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture. Report of a study made under the Research and Marketing Act of 1946.

[b] For prunes, the older procedure of finally adding 65 ml. of acid instead of 30 ml. and a standing time of 10 minutes instead of 4, was used. This was done before the present procedure was developed, but the latter would have given equal moisture values *(2)*.

[c] Further details are given in reference *(2)*.

TABLE 1
Precision of the dichromate oxidation procedure

Materials	Number of replicates	Range of specimen weights	Moisture content	Dichromate volume per g. of moist specimen	Standard deviation from mean	Standard deviation in terms of moisture content
		g.	%	*ml. per g.*	%	%
Pineapple-rice pudding	12	0.6788 — 0.7907	67.7	24.24 ± 0.04	0.28	0.09
Rice	10	0.2435 — 0.2595	10.1	71.03 ± 0.07	0.13	0.11
Prunes	12	0.9832 — 1.3163	19.0	12.82 ± 0.02	0.20	0.16
Corn	10	0.7909 — 0.8806	72.3	21.90 ± 0.06	0.30	0.08

Calculation. The moisture content is calculated as follows: Moisture % equals 100 $(1 - F \times D)$, where F is the "dichromate factor" in grams of dry material per ml. of dichromate and D is the milliliters of dichromate consumed per gram of specimen. F is determined from the equation by use of the reference moisture value for percent moisture.[4]

PRECISION

The reproducibility of the oxidation procedure was found to be very high in the previous publication *(2)*. With 10 to 12 replicates, standard deviations from the mean of 0.10% for potatoes, 0.13% for peas, 0.12% for glucose, and 0.09% for sucrose were obtained. Similar information was obtained for the present materials by oxidation of 10 to 12 presumably identical specimens of each, by the technique described. The results, given in Table 1, show that the mean deviations for pineapple-rice pudding, rice, prunes, and corn, were 0.28, 0.13, 0.20, and 0.30%, respectively. These standard deviations corresponded to moisture errors of 0.09, 0.11, 0.16, and 0.08%, respectively.

ACCURACY

The accuracy of the method is defined here as the agreement between moisture values, calculated by the use of mean dichromate factors, and moisture values by the reference method (however arbitrary) regardless of composition. Therefore moisture error is the difference between the value calculated by the use of the mean dichromate factor and the value by the reference method.

Pineapple-rice pudding. Work with this commodity was undertaken in cooperation with the Quartermaster

[4] Further details are given in reference *(2)*.

TABLE 2
Dichromate factor F and moisture results for various samples of pineapple-rice pudding

Batch numbers	Dichromate factor F	Moisture content		
		Vacuum oven	Calculated [5]	Error
	g. (dry) per ml.	%	%	%
21	0.01322	67.9	67.6	0.3
17	0.01325	67.3	67.1	0.2
5	0.01325	68.1	67.9	0.2
4	0.01326	68.4	68.2	0.2
23	0.01326	67.8	67.6	0.2
5a	0.01328	67.2	67.1	0.1
10	0.01330	67.9	67.8	0.1
1	0.01331	67.8	67.8	0.0
9	0.01331	67.7	67.7	0.0
13	0.01331	67.7	67.7	0.0
15	0.01331	67.8	67.8	0.0
6	0.01332	66.4	66.4	0.0
16a	0.01332	67.5	67.5	0.0
20	0.01332	68.0	68.0	0.0
22	0.01333	67.4	67.4	0.0
24	0.01333	68.0	68.0	0.0
2	0.01335	66.6	66.6	0.0
11	0.01335	67.7	67.7	0.0
12	0.01335	68.7	68.7	0.0
18	0.01337	68.1	68.2	0.1
7	0.01338	68.1	68.2	0.1
25	0.01339	67.2	67.3	0.1
16	0.01340	67.5	67.7	0.2
26	0.01341	67.8	68.0	0.2
8	0.01344	67.3	67.6	0.3
3	0.01345	66.2	66.5	0.3
Means:	0.01333	67.6		0.10
Standard deviation:	±0.44%			

[5] Moisture values calculated using mean F = 0.01333 g. per ml. for all samples.

TABLE 3
Dichromate factor F and moisture results for various samples of rice

Sample No.	Variety	Dichromate factor F	Moisture content		
			Vacuum oven	Calculated [6]	Error
		g. (dry) per ml.	%	%	%
		White rice [7]			
54	Nira	0.01273	11.8	12.4	0.6
78	Rexoro	0.01268	11.4	11.7	0.3
81	Blue Bonnet	0.01268	11.9	12.2	0.3
1	Cal Rose	0.01267	13.5	13.7	0.2
2	Mochigome	0.01267	12.4	12.6	0.2
3	Blue Bonnet	0.01265	10.4	10.5	0.1
4	Calif. Pearl	0.01261	11.0	10.8	0.2
38	Ark. Pearl	0.01260	9.6	9.3	0.3
74	Patna	0.01259	12.6	12.3	0.3
75	Pearl	0.01256	13.0	12.4	0.6
	Means:	0.01264	11.8		0.31
	Standard deviation:	±0.41%			
		Brown rice [7]			
25	Pearl	0.01250	13.9	14.1	0.2
11	Pearl	0.01247	14.8	14.8	0.0
33	Pearl	0.01246	13.3	13.2	0.1
19	Cal Rose	0.01246	13.1	13.0	0.1
	Means:	0.01247	13.8		0.10
	Standard deviation:	±0.15%			
		Paddy rice [7]			
5	Cal Rose	0.01329	13.5	14.3	0.8
6	Patna	0.01316	10.4	10.3	0.1
7	Pearl	0.01313	12.6	12.3	0.3
8	Blue Bonnet	0.01309	10.5	10.0	0.5
	Means:	0.01317	11.7		0.42
	Standard deviation:	±0.66%			
		Pre-cooked rice [7]			
9	Patna	0.01263	54.5	54.9	0.4
10	Brand A	0.01262	63.3	63.6	0.3
12	Brand B	0.01254	9.6	9.6	0.0
13	Quick Rice	0.01253	7.3	7.3	0.0
14	Brand C	0.01252	73.3	73.3	0.0
15	Pearl	0.01251	47.3	47.2	0.1
16	Patna	0.01250	58.2	58.1	0.1
17	Brand D	0.01248	73.8	73.7	0.1
18	Brand E	0.01243	63.5	63.2	0.3
	Means:	0.01253			0.14
	Standard deviation:	±0.51%			

[6] Calculated for each class of rice using the mean dichromate factor for that class.

[7] Rice as it is harvested, that is, with husks and bran present, is called paddy rice. Brown rice is obtained by removing just the husk and leaving the bran on the kernels. When both the husk and the bran are removed from paddy rice, white rice results. Two types of white rice are included in the table, milled and milled-parboiled, both types giving essentially the same dichromate factor. The milled-parboiled rice differs from milled rice in that the former is steeped in water for about 1 hour, autoclaved for 15 minutes at 15 p.s.i.g., then dried to approximately 15% moisture before milling to remove the husk and bran. The pre-cooked samples included commercial and experimental samples of canned white rice.

Inspection Division, Oakland Quartermaster Procurement Agency, Oakland, Calif. The manufacturer of the product had difficulty in meeting moisture specifications due to the lack of a rapid control moisture method.

The purchase specifications *(4)* called for 68.0 ± 0.5% moisture, as determined by 18 hours of vacuum oven drying, obviously unsuited for control purposes.

The dichromate method offered the possibility of obtaining moisture results in a few minutes, based upon the 18-hour-vacuum oven procedure. The essential question, however, was whether or not the oxidation factor would vary prohibitively with the ordinary variations in composition of such a heterogeneous product,[e] as permitted by commercial tolerances. The extent of such variations was investigated by analyzing 26 samples of the product, representing different batches, obtained from the manufacturer.

The variations in dichromate factors are shown in Table 2, Column 2. The percent moisture errors shown in Column 5 are all within the tolerance limits (±0.5% moisture) of the specifications, with a mean error of 0.10% moisture. The method was used successfully as a control method by the manufacturer.

Rice. The variation in dichromate factor with rice samples is shown in Table 3, Column 3. These 27 samples, which differed in variety and processing, included milled rice, milled-parboiled rice, brown rice, paddy rice, and both dry and moist pre-cooked canned rice. The results, of course, show the effect due to the type of processing used. Milled and milled-parboiled rice gave essentially the same dichromate factors, but the dichromate factors for brown rice were considerably lower, because bran was present. Paddy rice gave a much higher dichromate factor, due to the presence of the husks which were only partially oxidized, as seen from the presence of pieces of unoxidized husk in the reaction mixture after oxidation. Despite this obviously incomplete oxidation, the agreement between individual F values for paddy rice demonstrates the reproducibility of the oxidimetric technique. In the case of the precooked rices, the loss of soluble constituents during the cooking process resulted in dichromate factors which were lower than those for uncooked white rices.

When mean dichromate factors for the 4 different types of rice were used to calculate moisture contents, the results given in Column 5 were obtained. The re-

[e] On a dry basis, the pudding consisted of 39.0% crushed pineapple, 10.1% pineapple juice, 20.5% sugar, 14.6% rice (dry), 4.4% whole egg, 4.1% whole milk solids, 3.8% lemon juice, 2.9% corn starch, and 0.6% salt.

TABLE 4

Dichromate factor F and moisture results for various samples of prunes

Sample No.	Variety	Location[1] of production	Crop year	Maturity	Dichromate factor F	Moisture content		
						Vacuum oven	Calculated[b]	Error
					g. (dry) per ml.	%	%	%
28	Imperial	Yuba City	1949	Firm-ripe	0.01360	16.6	15.3	1.3
15	French	Yuba City	1950	Green-ripe	0.01361	19.2	17.9	1.3
4	French	Yuba City	1947	Green-ripe	0.01361	18.9	17.7	1.2
30	Imperial	Yuba City	1949	Green-ripe	0.01364	17.6	16.5	1.1
35	French	Santa Clara	1949	Green-ripe	0.01368	17.8	17.0	0.8
38	Imperial	Alexander Valley	1949	Green-ripe	0.01369	15.7	14.9	0.8
37	Imperial	Alexander Valley	1949	Overripe	0.01370	16.2	15.5	0.7
1	French	Hamilton City	1949	Firm-ripe	0.01373	17.1	16.6	0.5
5	French	Yuba City	1947	Overripe	0.01374	22.1	21.6	0.5
13	French	Yuba City	1950	Overripe	0.01375	25.3	24.9	0.4
12	French	Yuba City	1948	Overripe	0.01377	12.2	11.9	0.3
41	Sugars	Alexander Valley	1949	Firm-ripe	0.01378	16.3	16.1	0.2
19	French	Santa Clara	1950	Firm-ripe	0.01378	19.0	18.8	0.2
39	Imperial	Alexander Valley	1949	Firm-ripe	0.01378	17.1	16.9	0.2
6	French	Yuba City	1947	Firm-ripe	0.01379	19.4	19.2	0.2
29	Imperial	Yuba City	1949	Overripe	0.01379	16.5	16.3	0.2
33	French	Alexander Valley	1949	Overripe	0.01379	14.0	13.8	0.2
21	French	Santa Clara	1950	Green-ripe	0.01379	20.3	20.1	0.2
11	French	Yuba City	1948	Green-ripe	0.01380	15.3	15.2	0.1
34	French	Santa Clara	1949	Firm-ripe	0.01380	17.9	17.8	0.1
31	French	Alexander Valley	1949	Green-ripe	0.01380	15.8	15.7	0.1
18	French	Napa	1949	Green-ripe	0.01381	17.8	17.7	0.1
10	French	Yuba City	1948	Firm-ripe	0.01382	15.9	15.9	0.0
25	Imperial	Santa Clara	1950	Firm-ripe	0.01383	17.4	17.5	0.1
14	French	Yuba City	1950	Firm-ripe	0.01384	21.0	21.1	0.1
42	Sugars	Alexander Valley	1949	Green-ripe	0.01386	16.3	16.5	0.2
24	Robes	Hamilton City	1949	Green-ripe	0.01387	16.1	16.4	0.3
9	Imperial	Yuba City	1950	Green-ripe	0.01388	17.0	17.4	0.4
32	French	Alexander Valley	1949	Firm-ripe	0.01389	15.5	15.9	0.4
20	French	Santa Clara	1950	Overripe	0.01389	20.0	20.4	0.4
7	Imperial	Yuba City	1950	Overripe	0.01389	19.9	20.3	0.4
16	French	Napa	1949	Firm-ripe	0.01390	17.7	18.2	0.5
26	Imperial	Santa Clara	1950	Overripe	0.01390	20.3	20.8	0.5
27	Imperial	Santa Clara	1950	Green-ripe	0.01391	19.1	19.6	0.5
8	Imperial	Yuba City	1950	Firm-ripe	0.01391	17.5	18.0	0.5
2	French	Hamilton City	1949	Green-ripe	0.01391	17.1	17.6	0.5
22	Robes	Hamilton City	1949	Firm-ripe	0.01392	17.3	17.9	0.6
17	French	Napa	1949	Overripe	0.01394	16.0	16.7	0.7
36	French	Santa Clara	1949	Overripe	0.01396	15.7	16.6	0.9
23	Robes	Hamilton City	1949	Overripe	0.01397	15.5	16.4	0.9
3	French	Hamilton City	1949	Overripe	0.01400	12.7	13.8	1.1
40	Sugars	Alexander Valley	1949	Overripe	0.01400	16.3	17.4	1.1
				Means:	0.01382	17.4		0.50
				Standard deviation:	±0.75%			

[1] All locations are in California.

[b] Calculated using the mean dichromate factor F = 0.01382 g. per ml. for all samples.

sulting moisture errors shown in Column 6 gave mean errors of 0.31, 0.10, 0.42, and 0.14% moisture for dry white rice, brown rice, paddy rice, and pre-cooked canned rice, respectively. In the case of the moist pre-cooked rices, use of the mean dichromate factor determined for uncooked white rice to calculate moisture contents, resulted in a moderate mean moisture error of 0.3% due to the high moisture content of these samples. The similarity of the dichromate factors for the dry white rices indicates that changes in the variety of rice used in the production of pre-cooked rices, or of such rice foods as pineapple-rice pudding, would not seriously affect the mean dichromate factor determined for these products.

Prunes. The 42 experimental prune samples obtained from the Food Technology Division, University of California, Berkeley, Calif., were studied and the individual dichromate factors determined are shown in Column 6 of Table 4. These samples included 4 varieties, 5 localities of production, 4 crop years, 3 maturity levels, and various processing procedures. Moisture content of the samples ranged from 12.2 to 25.3%, the lower values being considerably drier than the normal commercial product.

The results show a trend in dichromate factor with maturity, the over-ripe samples tending to give high values, the green-ripe samples tending to give low values and most of the firm-ripe samples giving intermediate values. Many of the green-ripe samples were rubbery in texture and probably were not acceptable commercially.

If the 42 prune samples studied could be considered representative of dried prunes, then the moisture content of any random sample can be determined by this method by use of the mean dichromate factor F equal to 0.01382 g. per ml. with a mean error of 0.50% moisture. The correctness of this conclusion was studied by determining the moisture content of 6 samples of prunes purchased in local shops, 3 samples of fresh prunes, and 7 samples of rehydrated experimental prunes, with application of the mean dichromate factor. The results given in Table 5 show that the moisture content of these random samples can be rapidly determined by the dichromate method with a mean error of 0.5% moisture.

Corn. The 23 samples of frozen sweet corn obtained from the Department of Vegetable Crops, Utah State Agricultural College, Logan, Utah, ranging in moisture

TABLE 5

Moisture errors for random prune samples upon application of a predetermined mean dichromate factor

Sample No.[l]	Description	Moisture content		
		Vacuum oven	Calculated[m]	Error
		%	%	%
43	Brand A, Santa Clara Prunes, large	20.1	21.0	0.9
44	Brand B, Santa Clara French Jumbo Prunes	23.0	22.8	0.2
45	Brand C, California Prunes, large	25.3	25.9	0.6
46	Brand D, large size prunes	20.7	21.3	0.6
47	Brand D, stewed dried prunes (in heavy syrup)	67.8	67.5	0.3
48	Brand E, Prunes, large size	27.2	27.5	0.3
49	Prunes, fresh, green-ripe	69.6	69.2	0.4
50	Prunes, fresh, firm-ripe	69.1	68.7	0.4
51	Prunes, fresh, over-ripe	67.1	67.0	0.1
52	Prunes, French, firm-ripe, 1949	33.4	32.8	0.6
53	Prunes, French, green-ripe, 1949	30.8	31.3	0.5
54	Prunes, Imperial, firm-ripe, 1950	37.6	38.3	0.7
55	Prunes, French, green-ripe, 1949	29.7	29.5	0.2
56	Prunes, Sugar, green-ripe, 1949	33.9	34.8	0.9
57	Prunes, French, firm-ripe, 1947	32.6	32.1	0.5
58	Prunes, French, firm-ripe, 1950	38.3	37.5	0.8
			Mean:	0.50

[l] Samples Nos. 43-48 were commercial samples purchased in local stores. Samples Nos. 49-51 were fresh samples obtained from Mt. St. Helena, California. Samples Nos. 52-58 were rehydrated experimental samples.

[m] Calculated using the mean dichromate factor F = 0.01382 g. per ml. (see Table 4) for all samples.

TABLE 6

Dichromate factor F and moisture results for various samples of corn[a]

Sample No.	Variety[o]	Maturity level[p]	Processing procedure[p]	Dichromate factor F	Moisture content		
					Vacuum oven	Calculated[q]	Error
				g. (dry) per ml.	%	%	%
2	Hybrid 9556	Medium	..	0.01312	80.8	81.4	0.6
1	Carmel Cross	1	1	0.01300	81.7	82.2	0.5
3	Golden Cross	3	1	0.01291	69.1	69.7	0.6
4	Carmel Cross	2	1	0.01286	78.7	79.0	0.3
6	Hybrid 394	Medium	..	0.01275	77.1	77.2	0.1
5	Golden Cross	Medium	..	0.01274	76.1	76.2	0.1
7	Golden Cross	1	1	0.01272	77.6	77.7	0.1
8	Hybrid 1824	Medium	..	0.01268	79.4	79.4	0.0
9	Carmel Cross	4	1	0.01267	69.9	69.9	0.0
10	Golden Cross	2	1	0.01265	72.0	71.9	0.1
11	Golden Crown	Medium	..	0.01264	75.9	75.8	0.1
12	Iochief	Medium	..	0.01263	79.0	78.9	0.1
16	Hybrid KFV 50-15	Medium	..	0.01262	77.8	77.7	0.1
17	Carmel Cross	3	5	0.01261	73.8	73.7	0.1
13	Golden Cross	4	1	0.01261	65.9	65.7	0.2
14	Carmel Cross	3	3	0.01261	72.2	72.0	0.2
15	Carmel Cross	3	1	0.01260	72.3	72.1	0.2
19	Hybrid J-5	Medium	..	0.01259	75.0	74.8	0.2
20	Carmel Cross	3	2	0.01258	73.8	73.6	0.2
18	Carmel Cross	3	6	0.01257	74.0	73.8	0.2
21	Seneca Chief	Medium	..	0.01255	73.9	73.6	0.3
22	Carmel Cross	3	4	0.01252	73.3	73.0	0.3
23	FM Cross	Medium	..	0.01251	78.5	78.2	0.3
			Means:	0.01268	75.1		0.21
			Standard deviation:	±1.2%			

[a] These samples had moisture contents approximately those of freshly picked corn. All samples were processed, vacuum packed in No. 2 cans, frozen and stored at —10° C.

[o] All samples were either commercial varieties or specially selected hybrids for commercial use.

[p] Maturity level and processing procedure are defined in reference *(3)*.

[q] Calculated using the mean dichromate factor F = 0.01268 g. per ml. for all samples.

content from 65.9 to 81.7% and including 11 varieties, 6 processing procedures and 4 maturity levels, were used in this study. The individual dichromate factors given in Column 5 of Table 6 show a trend with processing procedure[f] in that all the samples, for which the procedures used resulted in higher losses in soluble solids, gave values which were lower than the mean. Use of the mean dichromate factor of 0.01268 g. per ml. to calculate moisture values results in a mean error of 0.21% moisture for these samples of corn.

SUMMARY

The rapid dichromate method for determining moisture content of fruits and vegetables has been applied to pineapple-rice pudding, rice, prunes, and fresh frozen corn, and the precision and accuracy of the method for these commodities have been determined. The reproducibility of the oxidimetric procedure for pineapple-rice pudding, rice, prunes, and corn was found to be, respectively, 0.28, 0.13, 0.20, and 0.30% standard deviation of 10 to 12 replicates from the mean. Analytical reproducibilities for moisture content were found to be, respectively, 0.09, 0.11, 0.16, and 0.08% standard deviation from the means of 10 to 12 replicates of each, for moisture contents of 67.7, 10.1, 19.0, and 72.3%, respectively.

[f] Processing procedures are defined in reference *(3)*.

A large number of samples of each, representing different batches, varieties, crops, maturity levels, and processing procedures, were studied. Moisture contents, calculated with mean dichromate factors, gave mean errors of 0.11, 0.30, 0.50, and 0.21% moisture, respectively.

Acknowledgments

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Flavor Components of Raspberries

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(Manuscript received May 24, 1952)

A REVIEW

The major work concerning analyses of the flavoring of raspberries is reviewed and the aromatic compounds isolated by various investigators are noted. Their relationship to the formulation of raspberry flavors is discussed. The aromatic compounds commonly recommended for commercial raspberry essences and oils are listed. The evidence concerning the presence or absence of β-ionone in natural raspberry flavor is considered. α-Irone, a new and now available aromatic chemical, is suggested for use in the preparation of commercial respberry flavors.

The flavor chemist and technologist is very often, if not always, interested in developing an imitation or an artificial flavor which simulates with a high degree of fidelity the natural flavoring material. In order to be able to do this adequately, it is a great help to have available the results of the analyses of the flavoring parts of the natural product. Unfortunately relatively little work has been done on this subject, or at least relatively little has been published for many fruits. There has been considerable work published on a few fruits, including raspberries and this is, of course, of great value as a guide for the formulation of raspberry flavor compositions.

However, one should not think that these isolated flavoring components necessarily were present in the fruit itself, even if precise methods were used for the separation. Because of the rather drastic treatment to which the fruit is subjected in attempting to recover the flavoring ingredients, there is every likelihood that decomposition products or derivatives of the original components have been obtained. For instance, when readily saponifiable esters are present, the analyst will probably recover the free acid and the free alcohol and one could not jump to the conclusion that these were present in the free state in the original fruit.

The more important work on the determination of the flavoring components of raspberry, *Rubus idaeus* L., is that of Coppens and Hoejenbos *(3)*, of Bohnsack *(2)*, and of Naves *(9)*. In the prior work done in this field, it should be noted that in 1929 Elze *(4)* claimed to have separated irone as the *p*-bromophenylhydrazone, anisaldehyde as the semicarbazone, nerol, and an undetermined paraffin melting at 43-44° C., from the

steam distillate of the oil obtained from raspberries. It is clear, however, from the meticulous work of Coppens and Hoejenbos, of Bohnsack, and of other investigators, that Elze could not have been working with the natural product for none of these investigators were able to isolate the aforementioned compounds. Elze probably was analyzing a material which contained some synthetic oil. Irone, nerol, and anisaldehyde had been used for years before 1929 in the formulation of commercial raspberry flavors.

EXTRACTING THE ESSENCE

As representative of the method of extracting raspberry essence, one may describe the procedure of Coppens and Hoejenbos. They pressed out 480 kg. of raspberry juice from 510 kg. of fresh raspberries. This juice was extracted with ether in a continuous extractor to yield an acid solution which was washed with 10% sodium carbonate solution to obtain the free acids as the sodium salts. After acidification, these were extracted with ether, which then was evaporated off. After steam distilling the residue, acetic, *n*-caproic, and benzoic acids were isolated and identified. Some other fatty acids were present in small amounts but were not identified.

The acid solution remaining after the ether extraction was neutralized and evaporated to yield a dark, pasty residue having a characteristic raspberry odor. This was distilled to yield four fractions.

Fraction I was shown to contain diacetyl, ethyl alcohol, and ethyl acetate. Fraction II also contained diacetyl, and isoamyl alcohol was identified as a part of this fraction. Fraction III was treated with sodium bisulfite to recover the aldehydes, and benzaldehyde was obtained in this manner.

Refractionation of fraction IV was performed to give 3 subfractions IVa, IVb, and IVc. Fraction IVa, upon addition of 3% sodium hydroxide solution saturation with carbon dioxide and the addition of a phenol reagent, gave reactions which were interpreted as being due to a salicylate. The rest of this portion was saponified, subjected to phthalization, and the aromatic alcohols benzyl and phenylethyl alcohol were obtained. Menthone was also found in this fraction along with an acid melting at 108-109° C., which was not identified but which was shown not to be benzoic, phenylacetic, or *m*-methoxybenzoic acid. Fraction IVb was shown to contain additional quantities of phenylethyl alcohol, coumarin, and some sesquiterpene hydrocarbons and alcohols which they did not identify. Coumarin was also found in the third subfraction, IVc, and this portion also contained a material, melting at 54.5-55.5° C. which they considered to be a saturated acid but which they could not identify.

AROMATIC COMPONENTS

A recapitulation of their work indicates that Coppens and Hoejenbos *(3)* found: 1, acids: acetic, *n*-caproic, and benzoic; 2, alcohols: ethyl, isoamyl, benzyl, and phenylethyl; 3, ketones and diketones: diacetyl and menthone; 4, aldehyde, benzaldehyde; and 5, ester, ethyl acetate. There were also indications that acetoin (acetylmethylcarbinol), coumarin, and a salicylate were also present. As mentioned above, they could find no evidence for the presence of anisaldehyde, irone, or nerol.

In 1941 Bohnsack *(2)* in an investigation principally directed towards detecting in strawberries the presence and evaluating the amount of 3-hexen-1-ol (β-γ-hexenol), more commonly known as leaf alcohol, also repeated much of the work of Coppens and Hoejenbos. In addition, however, Bohnsack prepared a number of derivatives of leaf alcohol which are of interest. Thus he oxidized 3-hexen-1-ol to oxalic acid and proprionic acid; he hydrogenated this alcohol to the *n*-hexanol; and prepared the formate boiling at 53-55° C. at 12 mm., the acetate boiling at 66° C. at 16 mm., and the isobutyrate boiling at 80° C. at 14 mm. The formate of 3-hexen-1-ol has a powerful cabbage odor; the acetate smells like the acetates of isoamyl and *n*-hexyl alcohols; and the isobutyrate has an odor similar to that of *n*-hexyl butyrate.

In addition then to the compounds found by Coppens and Hoejenbos, Bohnsack found that isobutyl alcohol and leaf alcohol were present in natural raspberry flavor.

McGlumphy *(7)*, without detailing the methods used, reported that he and his co-workers identified the following as constituents of black raspberries: 1, acids: formic, acetic, benzoic, and hydrocinnamic; 2, alcohols: ethyl, butyl, isobutyl, isoamyl, and benzyl alcohols, and menthol; 3, ketones and diketones: acetone, diacetyl, and menthone; 4, aldehydes: acetaldehyde, isobutyraldehyde, and benzaldehyde; 5, esters: ethyl acetate, ethyl acrylate, and ethyl salicylate; and also catechol. They were not able to confirm the presence of coumarin or phenylethyl alcohol as reported by Coppens and Hoejenbos, nor of anisaldehyde, irone, and nerol as reported by Elze, nor of β-ionone as indicated by Bohnsack which is discussed below.

Volatile aromatics recovery. The Eastern Regional Research Laboratory has developed a process *(5, 8, 14)* for the recovery and subsequent concentration of the more volatile components of the flavoring matter of fruits. This was initially developed for apples and was later applied to grapes and other fruits. It is likely that with the aid of this process, it will be possible to trap and thus identify the highly volatile components of fruit flavors.

IMITATION RASPBERRY FLAVORS

A survey of many of the published formulas for imitation raspberry flavors made by Jacobs *(6)* indicates that some 35 compounds have been recommended for incorporation into such compositions. Among these are 1, esters: the methyl, ethyl, propyl, isobutyl, isoamyl, cyclohexyl, and benzyl acetates; the ethyl, isobutyl, and isoamyl formates; the methyl, ethyl, isoamyl, and cyclohexyl butyrates; the methyl, ethyl, and benzyl benzoates; and isoamyl isovalerate, ethyl enanthate, ethyl sebacate, ethyl salicylate, and methyl cinnamate; 2, alcohols: phenylethyl alcohol, geraniol, and linalool; 3, acids: tartaric and succinic; and 4, aldehydes: benzaldehyde and cinnamaldehyde; and also butyl β-naphthyl ether, bromelia, eugenol, γ-nonyl lactone, vanillin, coumarin, and β-ionone.

In addition to these, Jacobs found that 32 other esters, 6 other alcohols, 3 other ketones, and 3 compounds of other functional groups have been suggested as suitable for incorporation into commercial raspberry flavors. Indeed there are few other flavors for which so many aromatics have been recommended or suggested.

Furthermore there are a number of other natural flavorings and essential oils which are useful and have been utilized for the preparation of raspberry essence. Among these may be mentioned orris oil, cinnamon oil, clove oil, jasmine oil, petitgrain, and rose oil.

β-Ionone. Wilson *(15)* in a comprehensive work concerning the identification of flavoring components of

commercial flavors, came to the conclusion that β-ionone was not a component of natural raspberry flavor and thus verification of the presence of β-ionone could be assumed to indicate that a product was an imitation flavor or that a product has been fortified with an imitation, or that an imitation flavor had been added to a natural flavor.

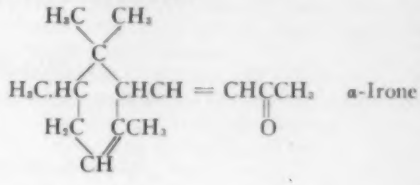
He preferred the use of *m*-nitrobenzhydrazide as the reagent for the determination of β-ionone and his method was adequate for the estimation of quantities of the order of 10 to 100 mg. of β-ionone. The sample (250-1000 g.) was steam distilled and 300 to 500 ml. of distillate was collected. Sufficient water was added to reduce the alcohol content to 10%, then the mixture was extracted with ether, then 95 to 100 mg. of *m*-dinitrobenzhydrazide and 0.2 ml. of acetic acid were added, and the mixture was evaporated on a steam bath while passing in a current of air to hasten the evaporation and prevent the temperature from rising. The evaporation was continued until 1 to 3 ml. of an either watery liquid and at times oily residue was obtained. This residue was dissolved in 5 ml. of alcohol, 5 ml. of water was added, and the mixture warmed to get a clear solution, if necessary and 0.2 ml. of acetic acid was added and the mixture was allowed to stand for 2 hours. Then 5 ml. of water was added drop by drop while continuously swirling the flask and the mixture was kept at room temperature for either 1 hour or overnight. It was then refrigerated overnight or for 48 hours, filtered on a fritted glass crucible of porosity 4, washed with about 30 ml. of dilute alcohol, and dried in a vacuum oven at 70° C. and weighed. The weight of the precipitate multiplied by the factor 0.541 is equivalent to β-ionone.

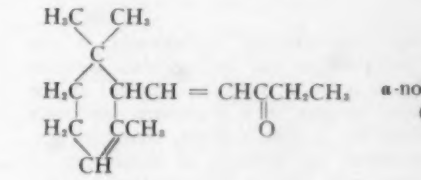
In 1947 Naves *(9)*, who had been working on the problem of establishing the presence of the ionones in various plant products and had found that β-ionone is present in the distillate of the concrete of brown boronia of Australia (*Boronia megastigma* Nees) *(10)*, disclosed that Bohnsack had informed him that in a study of an essence obtained from the marc of raspberries he (Bohnsack) had isolated a very small amount of β-ionone which was identified by means of its semicarbazone, melting at 148-149° C. The melting point did not change when a mixed melting point with the synthetic material was determined. When this carbazone was boiled with an aqueous solution of oxalic acid, the characteristic odor of β-ionone could be clearly distinguished.

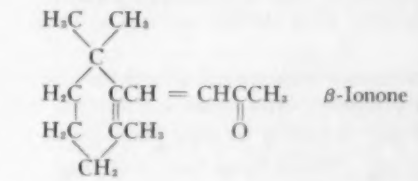
Naves remarked that Bohnsack thought that the β-ionone arose as a degradation product of carotene or of some related compound. Naves pointed out that ionone often results as the oxidation products of carotenoids or when they are subjected to other treatment such as exposure to ultraviolet light. Thus Naves stressed that the work of Bohnsack raised the question again, despite the work of Wilson, as to whether or not β-ionone is present in raspberries. The work of McGlumphy *(7)* as noted above did not reveal the presence of β-ionone in raspberries.

α-Irone. Within the past year a highly purified α-irone has been made commercially available *(1)* as the result of the work of Naves and his co-workers in synthesizing this compound *(11, 12, 13)*. The production of this aromatic chemical has made a new material available to the flavor chemist.

α-Irone is an isomeric methylionone and should not be confused with the methylionones such as the Raldeines. The differences in structure can be readily seen from the following formulas:


α-Irone


α-normal Methylionone (Raldeine type)


β-Ionone

It can be seen then that β-ionone is 4-(2, 6, 6-trimethyl-1-cyclohexenyl)-3-buten-2-one; α-normal methylionone, also known as cyclocitrylidene ethyl methyl ketone is 5-(2, 6, 6-trimethyl-2-cyclohexenyl)-4-penten-3-one; α-irone may be considered as a 4-(2, 5, 6, 6-tetramethyl-2-cyclohexenyl)-3-buten-2-one.

Among the fruit essences for which α-irone is considered particularly suitable is raspberry flavor, α-Irone has several advantages over orris as a flavor component. It is a chemically pure substance and thus is uniform from lot to lot. It does not have the bitterness of orris and it does not have too much floral character of an undesirable type for flavors. It is likely that α-irone will be found useful in giving raspberry flavor a new and desirable note.

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Onion Pungency and Onion Flavor: Their Chemical Determination[a]

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There are two characterizing components of the onion. 1. The lachrymating and bactericidal. Because of its instability, it must be promptly distilled at low temperature under vacuum by a procedure previously outlined to isolate it. 2. The specific, characteristic, flavorsome component, the potency of which can be varied about seven-fold, can be quantitatively determined at any potency by a method herewith outlined.

There seems to be no standardized acceptance of what constitutes onion pungency and what constitutes onion flavor. It is probably accurate to state that onion pungency to most people means the impression the onion gives in its raw state either as it is being peeled and trimmed, sliced, chopped, diced or otherwise prepared, or eaten directly, as in salads, sandwiches, etc.

If that is accepted as onion pungency, then its pungency should be evaluated by a determination of the chemical component responsible for that impression. Unquestionably the onion component that under the above conditions affects the senses most is the lachrymating principle. It has been identified *(3)* as a thioaldehyde and a method has been outlined for its isolation and identification.

The greatest use of the onion is to flavor many food preparations; sausages and many other meat concoctions; stews; soups; etc. The lachrymating principle, due to its instability, generally contributes little flavor to the types of food mentioned. If and when it does it is probably more likely to be unfavorable than favorable. Thioaldehydes, and as has been shown, the lachrymating principle of onion *(3)* rather quickly polymerize and become more or less inert. Under some conditions the lachrymating principle, because of its reactivity before polymerization, may give rise to unexpected flavors, flavors which have been characterized as garlic-like or skunky.

The component responsible for the characteristic onion flavor, as indicated by Semmler *(8)*, is allyl propyl disulfide. As will be shown, this occurs largely in a combined form in sound onion tissue but it is liberated by enzymes as the raw cell is ruptured. Neither the chemical structure nor the flavor of the combined form of this disulfide is known. Allyl propyl disulfide can be separated from the rest of the onion by steam distillation under suitable conditions and its sulfur is readily oxidized either to the sulfone stage or completely to sulfate by bromine. This offers a basis for its chemical estimation. The combined form in which allyl propyl disulfide exists in the sound onion tissue is non-volatile and its flavor is not known. Until it has been isolated it is impossible to know whether the mild but characteristic onion flavor of the whole cooked onion is due to a small amount of free allyl propyl disulfide, or to the combined form.

Several investigators *(2, 6, 7)* have suggested methods for determining onion flavor potency, but they all resolve themselves into a determination of all the sulfur that can be volatilized after hydrolysis in strong acid. This will include sulfur not in the flavorsome component. None of the methods take into account that the flavor potency of two similar lots of onions, or even the two halves of an onion, may, as one wills, be varied about seven-fold. The method described below makes possible the determination of the relative potency of the flavor in whichever strength within the possible several-fold variations it may be desired to use the onion. It is applicable to dehydrated onion as well as to fresh onion. To the degre that the dehydration does not significantly inactivate the enzymes or alter their substrate, the potency may be varied as with fresh onion. Sulfur-bearing amino acids give rise to hydrogen sulfide upon being heated. In the method to be described the hydrogen sulfide yielded by onions introduces a slight error but it is so small as to be insignificant.

The basis for the several-fold variation is that only a small amount of allyl-propyl disulfide occurs in the free state. The maximum possible amount is liberated by a thorough disintegration of the raw onion as with a hammermill type of machine. Slicing, coarse-grinding, chopping, dicing, etc. permit the enzymes to liberate intermediate amounts in proportion to the degree of the disintegration of the raw onion cells. Disintegration of dehydrated onion may be done in the dry state or after rehydration. This seems to be a unique instance in which a chemical determination can be made of a flavor that may be varied over such a wide range.

EXPERIMENTAL

The method is based on steam distilling the allyl propyl disulfide at 15 p.s.i.g. [250° F. (121° C.)] and with bromine oxidizing the sulfur in the distillate to sulfate, which can be then determined gravimetrically or colorimetrically *(5)*. While steam distillation at atmospheric pressure is an established procedure, it seems not to have been explored at elevated pressures for laboratory use. Some of its advantages have been previously indicated *(4)*. This is illustrated in Table 1 from which it is evident that C_6H_5Cl should be steam distilled at atmospheric pressure [212° F. (100° C.)] while C_6H_5I is more advantageously steam distilled under pressure at higher temperatures.

Such figures for any substance, A, can be calculated with the following formula: Weight of A in distillate divided by the weight of water in distillate equals molecular weight of A times vapor pressure of A at temperature T divided by the molecular weight of water times the vapor pressure of water at temperature T.

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 11, 1952.

TABLE 1

Efficiency of steam distillation at different temperatures

Substance	Amount of substance in distillate when distilled at				
	100° C.	120° C.	150° C.	200° C.	230° C.
	g. per liter H_2O	*g. per liter H_2O*	*g. per liter H_2O*	*g. per liter H_2O*	*g. per liter H_2O*
Iodobenzene, C_6H_5I	750	810	870	960	1000
Bromobenzene, C_6H_5Br	1620	1610	1580	1540	1530
Chlorobenzene, C_6H_5Cl	2410	2280	2100	1880	1780

Steam distillation of allyl propyl disulfide at atmospheric pressure [212° F. (100° C.)] is decidedly ineffective but under 15 p.s.i.g. [250° F. (121° C.)] it is quite effective.

Figure 1 illustrates the equipment in which the steam distillation under pressure and at elevated temperature [250° F. (121° C.)] is made. The flask *E* is made of a heavy Pyrex 1-liter erlenmeyer flask by sealing the mouth and fusing a ring neck into an opening in its bottom. The constriction *C* should be approximately 1/16 inch in diameter, but more particularly of a size to deliver 450 ml. distillate in 30 minutes with a retort temperature of 250 ° F. (121° C.). The enlarged forepart of *C* is to avoid particles or droplets being carried over by the distillate.

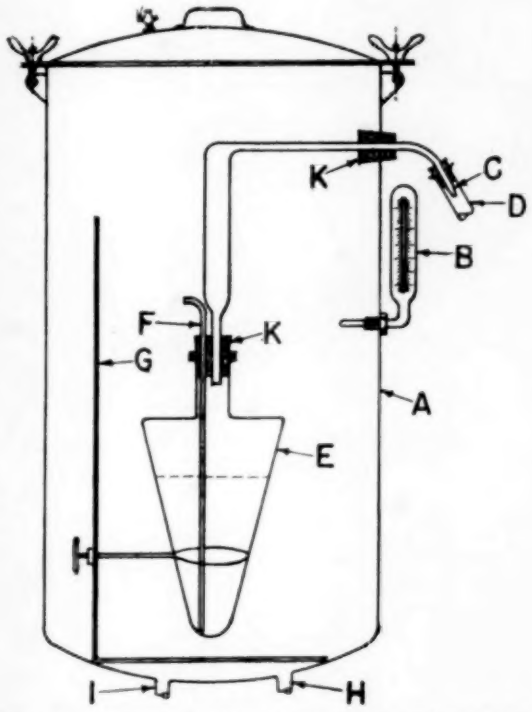

Figure 1. Diagram of steam distillation equipment for determination of allyl propyl disulfide.

A, retort or autoclave; B, thermometer; C, glass tube with constricted nozzle about 1/16 inch in diameter; D, pyrex bulbous 600 mm. condenser; E, special pyrex flask; F, steam inlet tube; G, ring stand; H, steam inlet; I, retort drain; and K, rubber stoppers.

Steam distillation under pressure has been found to be a useful tool in a variety of problems. Some raw products tend to froth. This can be prevented by first heating them to boil slightly before subjecting them to steam distillation under pressure. In case of volatiles that might escape, the preliminary heating to boil should be done under reflux. In case of liquids without pulp it is sometimes desirable to supply it with limited bits of filter paper to prevent droplets being carried over.

For determining onion flavor potency, a 200 g. sample of onion in the exact condition in which it is intended to use the onion is introduced into the flask with 200 ml. of water. The dilution with water avoids channeling by the steam when considerable pulp is present.

When all is in place, the retort cover tightened, the steam is turned on with the drain and the exhaust petcock left wide open to allow the air to escape through it rather than through the flask containing the onion. When the thermometer reaches preferably a few degrees above 212° F. (100° C.), the drain is closed and the temperature rapidly raised to 250° F. (121° C.). This can be attained in less than 2 minutes and if the nozzle is gauged to deliver about 450 ml., the next 30 minutes will bring over all but negligible proportions of the organoleptically detectable onion flavor. The distillate is collected in 50 ml. of bromine water containing a few drops of undissolved bromine. The excess bromine is boiled off and the boiling is continued to reduce the volume to about 150 ml. for convenient sulfate precipitation. The solution is then filtered, the sulfate precipitated with barium chloride, and the barium sulfate is determined either gravimetrically or colorimetrically *(5)*.

The results with the method compared with similar data obtained by the all-glass Long Tube Evaporator of Bartholonew *(1)* serves both to indicate the ineffectiveness of distillation at atmospheric pressure and illustrates a gross defect of the Long Tube Evaporator for fractional distillation. Onion juice was prepared by pressing ground onions with the Carver press. The results are given in Table 2.

TABLE 2

Comparison of results obtained using the Long Tube Evaporator and the present experimental method

Method	Sample	Distillation		
		Time	Volume	$BaSO_4$
	g.	*minutes*	*ml.*	*g.*
Long Tube	1000	53	350	0.1531
Long Tube	1000	38	206	0.0932
Experimental	250 plus 150 ml. H_2O	25	522	0.1137
Experimental	250 plus 150 ml. H_2O	25	510	0.1175
Experimental [b]	250 plus 150 ml. H_2O	25	511	0.1140

[b] This sample was taken from the combined two residues from the Long Tube experiments.

It is evident that the Long Tube Evaporator had no effect in concentrating the sulfur-bearing component in either the residue or in the distillate. Close observation of the Long Tube Evaporator offers an explanation for this performance. The narrow tube, about one fourth filled, offers but little surface area for the volatiles in the liquid to escape. Periodically the liquid becomes super-heated and then the whole column froths up and a portion boils over. Momentarily this should give the more volatile components an opportunity to escape and pass over to the vapor condenser. When the liquid then settles down, it leaves spread over the inner surface of the tube a film. As this is evaporated, it simulates the effect on a drum drier and therefore evaporates all volatiles, including water. The dried film thus deposited on the walls of the tube in the case of some materials often becomes difficult to remove.

To further show the inadequacy of low temperatures even with a greater amount of distillate, the aperture *C* in Figure 1 was restricted to yield only 200 g. distillate in 30 minutes at 250° F. (121° C.). The aperture was then enlarged and yielded 362 g. distillate in 30 minutes at 214° F. (101° C.). The distillates yielded, respectively, 0.0255 g. and 0.0137 g. of $BaSO_4$ with whole

cooked onions (ground after cooking), and 0.1385 g. and 0.0916 g. of $BaSO_4$ with the onions ground in a kitchen fruit and vegetable grinder using a plate with holes ⅛ inch in diameter. The samples used were in each case 400 g., consisting of equal weights of onion and water. Direct distillation in an hour yielded, respectively, 330 g. of distillate with 0.0174 g. of $BaSO_4$, for the whole cooked onions and 340 g. distillate with 0.1201 g. of $BaSO_4$, appreciably less than by the new method, which, with the aperture *C* restricted, was not intended to be quantitative.

The following interesting experiment was made with two varieties of commercial onions in the spring of the year. One variety was a new crop of Bermuda onions grown in Texas; the other was New York Yellow Globe onions from storage. Each onion was cut in 3 comparable sectors to obtain 3 similar samples for each variety. The three lots were treated as follows:

1. The onions were heated to boil in an equal weight of water and then simmered for 20 minutes under reflux. Reflux was provided by placing a stew pan containing cold water on the kettle in which the onions were simmered. After cooling the mass the onions were disintegrated with the high speed of a Waring blendor for about 15 seconds.

2. The onions were ground in a fruit and vegetable grinder using a plate with holes ⅛ inch in diameter, then heated to boil in an equal weight of water and simmered for 20 minutes under reflux. After cooling the mass it was further disintegrated with the high speed of a Waring blendor for about 15 seconds.

3. The onions were ground in the fruit and vegetable grinder and then disintegrated further with the high speed of a Waring blendor for about 20 seconds. They were then heated to boil in an equal weight of water and simmered for 20 minutes under reflux.

Each sample of 400 g. containing 200 g. of onion, was subjected to steam distillation for 25 minutes at 250° F. (121° C.) by the present method and then sulfate in the distillate was determined. The results are shown in Table 3.

TABLE 3

Comparison of amounts of barium sulfate obtained from two different varieties of onions prepared by different methods

Variety	Sample No. 1		Sample No. 2		Sample No. 3	
	Distillate	$BaSO_4$	Distillate	$BaSO_4$	Distillate	$BaSO_4$
	g.	g.	g.	g.	g.	g.
Bermuda	464	0.0111	464	0.0385	464	0.0926
Yellow Globe	473	0.0205	473	0.1095	473	0.1640

It is noted that the amounts of the distillates from the Bermuda onions were all identical (464 g.). The distillates of the Yellow Globe onions were also identical (473 g.). While the distillates should be identical if everything is exactly repeated in each analysis, they require identical coming up time and close regulation of the retort temperature. Somewhat more or less distillate collected at the end of the distillation period is of little significance since it contains but little of the allyl propyl disulfide. It is believed that the distillates from the Yellow Globe onions were slightly higher because they were obtained on a different day, the samples of Yellow Globe being determined against a barometric pressure of only 29.7 inches while those from the Bermuda onions were obtained against a barometric pressure of 30.2 inches.

The question arises as to the source of the small amount of sulfate in the No. 1 samples. Perhaps it represents free allyl propyl disulfide that exists in the sound onion tissue or freed as a result of the cells being ruptured in trimming and sectioning and otherwise bruising raw cells during handling.

An interesting observation unrelated to the analytical method in connection with the above experiment is that during the steam distillation there was very little browning of the whole cooked samples. There was much more in the No. 2 samples and far more in the No. 3 samples. In each comparable sample there was far more browning with the Yellow Globe onions than with the Bermuda onions. Evidently enzymes had released not only allyl propyl disulfide but also some other compound that is involved in non-enzymic browning during the heating period. The question arises as to whether this may be a sugar with which the allyl propyl disulfide was in combination as a glucoside. This might be a case of non-enzymic browning for which previous enzyme activity is responsible.

SUMMARY

The activity of enzymes is of much importance in the handling of fruits and vegetables. In the onion, the results of such activity can be followed chemically more readily than usual because the characterizing components of the onion are volatile and sulfur bearing and the sulfur is readily oxidized by bromine to sulfate and thus can be quantitatively determined. There is, in a free state, at most only a small amount of the characteristic flavorsome component, allyl propyl disulfide, in sound onion tissue. It is shown how enzymes may be employed to greatly increase it. A method is described to separate it from the onion by a special method of steam distillation under pressure and estimate it quantitatively. It therefore can be used to measure the flavoring ability of different varieties of onions and of each variety under any condition of its possible several-fold variation in flavor potency.

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Heat Inactivation of Pectinesterase in Citrus Juices[a]

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Data are presented to show the effect of varying temperatures and holding times on the percent inactivation of pectinesterase in Duncan grapefruit and Valencia orange juices containing 5 and 10% pulps. The influence of pH at three levels on the heat inactivation of the enzyme in the two juices at 10% pulp is also presented.

Desirable physical characteristics of good processed citrus juices and concentrates, when the latter are reconstituted, are that they retain the "cloud" of fresh juices and remain free of gel-like lumps. The cloud is retained and the formation of gel lumps is prevented by protecting the natural pectin found in the extracted products from enzymic deesterification and degradation by pectinesterase.

Both grapefruit and orange juices contain a sufficient amount of pectinesterase to demethylate the pectin during or after processing unless heat is applied to inactivate this enzyme. This is especially true if frozen citrus concentrates, with high pulp content and therefore increased activity, are not kept frozen. The use of heat is a practical means at present by which the processor may insure his product against gelation and clarification. However, such a preventive method may lower the quality of the product by affecting the flavor if the heat treatment applied is not carefully controlled and kept at a minimum to avoid changes in flavor.

Most of the studies on heat inactivation of pectinesterase reported in the literature, where results are indicated on the percentage of inactivation, have been made on products other than citrus.

In a study of tomato pectinesterase, Kertesz *(3)* reported on the heat inactivation and sensitivity of the enzyme to heat with varying pH. For tomato the pectinesterase is completely inactivated by heating the juice to 176° F. (80° C.) for 45 seconds. Holden *(1)* has shown that pectinesterase, extracted from tobacco leaves, heated in a phosphate buffer at pH 8.0 for 5 minutes at 158° F. (70° C.), 176° F. (80° C.), and 196° F. (91° C.) showed a 30% loss of activity at 158° F. (70° C.) and complete inactivation of pectinesterase at 176° F. (80° C.). Pollard and Kieser *(6)* extracted apple pectinesterase and reported the heat inactivation of the purified enzyme preparation at pH 3.5 and temperatures of 149° F. (65° C.) and 154.5° F. (68° C.). After heating for only 5 minutes at these two temperatures there was practically complete inactivation. Their results on inactivation of the enzyme in the raw apple juice were markedly different from those found for the purified enzyme preparation. Apple juice heated to 154.5° F. (68° C.) for 40 minutes retained 80% of the enzyme activity and a temperature of 176° F. (80° C.) for 15 minutes was necessary for complete inactivation.

Joslyn and Sedky *(2)* published data showing the effect of heating citrus juices in order to maintain a cloud. Valencia orange juice will maintain a cloud for 513 days when heated at 180.5° F. (82.5° C.) for over 10 minutes, at 185° F. (85° C.) between 8 and 10 minutes, at 188.5° F. (87° C.) for 2 minutes, and to a temperature of 194° F. (90° C.) at the natural acidity of the juice, pH 4.2. For both orange and grapefruit juice at pH 4.0, a heating period of 1 minute was required at 176° F. (80° C.), while at pH 2.5 the temperature required was 167° F. (75° C.). Stevens *(7)* described conditions whereby citrus juice may be stabilized for a long period. The temperature range for accomplishing this stabilization varied from 161.5° F. (72° C.) to 192° F. (89° C.) for 0.1 to 3 minutes with a range of pH 2.2 to 3.79, respectively. More recently, Stevens, Pritchett, and Baier *(8)* reported that at pH 3.8 satisfactory stabilization of deaerated orange juice of average orange pulp content could be accomplished at 192° F. (89° C.) with a 2-minute holding time. As the temperature was raised to 201° F. (94° C.) the holding time could be decreased to 15 seconds. With a pH of 3.3 the temperature required for stabilization was 185° F. (85° C.) for 2 minutes and 194° F. (90° C.) for 15 seconds. For grapefruit juice at pH 3.2 a temperature of 183° F. (84° C.) was required with a 2-minute holding time and 192° F. (89° C.) for 15 seconds.

This investigation was concerned with the effect of varying temperatures and holding times on the heat inactivation of pectinesterase found in Duncan grapefruit and Valencia orange juices and the effect of pH upon the inactivation by heat of pectinesterase in these juices. The results of this investigation are presented and discussed in this paper.

EXPERIMENTAL PROCEDURE

Preparation of samples. Juices from both Duncan grapefruit and Valencia oranges were extracted by the Citro-Mat extractor and run through a Food Machinery finisher (Model 35) with an 0.020 inch screen. The pulpy residue remaining was again run through the Food Machinery finisher with an 0.125 (⅛) inch screen. The two screened juices were separately passed through a Weil centrifugal turbine pump in order to cut the pulp particles to uniform size so that approximately 95% of the pulp passed an 0.0232 inch Tyler standard screen. Proportional amounts of the 0.020 inch screened juice were added to the 0.125 inch screened pulpy juice so that the resulting blended mixtures contained 5 and 10% pulp by volume as determined by the centrifugal method. These prepared juices, containing 5 and 10% pulp, were placed in 10 gallon drums, frozen and stored at —8° F. (—22° C.) until needed. The 5%

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[c] Florida Citrus Commission.

pulp level juice was equivalent to the pulp content usually found in commercial frozen concentrates.

Heat treatment. The juices, after being thawed and warmed to room temperature, were pumped through a pasteurizer containing a heating coil 15 feet in length and composed of 1/16 inch (internal diameter) stainless steel tubing. Hot water was circulated around the heating coil. The flow rate was adjusted to 560 ml. per minute, which indicated that it was in the turbulent flow range. The juice was in contact with heat in the pasteurizer for 0.8 second, the time required to raise the juice from room temperature to the desired temperature, herein referred to as the initial temperature. To hold the juice for any given time at a definite temperature, the juice was run directly from the pasteurizer into test tubes submerged in a holding bath regulated to the desired temperature. All samples were cooled instantly by flashing under vacuum into small receptacles. Temperatures were controlled by a Brown potentiometer pyrometer and checked with a Bureau of Standards certified thermometer.

Method of analyses. The method used for the measurement of pectinesterase activity in citrus juices included features from the procedures by MacDonnell, Jansen, and Lineweaver *(4)* and McColloch and Kertesz *(5)*. A Fisher Titrimeter was used to observe the pH. The substrate consisted of 50 g. of a 1.0% solution of citrus pectin containing 0.2 *M*. sodium chloride. To this substrate a 1 to 5 ml. sample of juice was added, mechanically stirred in a 150 ml. beaker, and rapidly titrated to pH 7.0 with 0.2 *N*. sodium hydroxide. The temperature was maintained at 30° C. for 30 minutes; this reaction time was preferred because many of the samples after heat treatment contained only traces of activity. During this reaction period the mixture was titrated with 0.05 *N*. sodium hydroxide to maintain a pH 7.0.

The pectinesterase units are expressed by the symbol (PE.u.) ml. per ° Brix, which represents the milliequivalents of ester hydrolyzed per minute per milliliter per degree Brix. Reducing the (PE.u.) ml. to one degree Brix is advisable for two reasons. Cooling the heated juice by flashing under vacuum changes the original concentration of the juice as well as the degree Brix, which is a measurement of soluble solids. By reducing to a common denominator such as degree Brix, a convenient means of expressing the change of concentration on a unit basis is derived for comparison. Also, "degree Brix" is rapidly measured by a refractometer and is a term commonly used and understood by citrus processors when expressing soluble solids.

The degree Brix of the juices was determined at 28° C. by using a Bausch and Lomb refractometer.

EXPERIMENTAL RESULTS AND DISCUSSION

Effect of varying temperatures and holding times on heat inactivation of pectinesterase found in Duncan grapefruit and Valencia orange juices. The original activity in the unheated grapefruit juice, expressed as (PE.u.) ml. per ° Brix, varied from 0.0164 to 0.0174 units for the 5% pulpy juice, while the grapefruit juice containing 10% pulp varied in activity from 0.0309 to 0.0373 units. Both pulpy juices had a pH of 3.5.

The temperatures to which both the 5 and 10% pulpy grapefruit juices were subjected in the pasteurizer ranged from 165° F. (74° C.) to 200° F. (93.5° C.) with holding times from 0 to 120 seconds. It is reiterated that the 0.8 second required to raise the juice from room temperature to the desired temperature was not a part of the holding time.

The effect of temperature and time on the inactivation of pectinesterase in Duncan grapefruit juices with 5 and 10% pulp is presented in Table 1. The actual (PE.u.) ml. per ° Brix and percentage of inactivation of the enzyme in these juices are shown in this table. The grapefruit juice had a pH of 3.5.

TABLE 1

Heat inactivation of pectinesterase in Duncan grapefruit juice (pH 3.5)

Temperature		Holding time	5% pulp		10% pulp	
			(PE.u.) per ° Brix	Inactivation	(PE.u.) per ° Brix	Inactivation
° F.	° C.	*seconds*	ml. × 10^3	%	ml. × 10^3	%
165	74.0	0	2.7	84.1	5.3	83.4
165	74.0	5	2.4	85.9	4.6	85.6
165	74.0	10	2.1	87.7	4.4	86.2
165	74.0	15	2.0	88.2	4.2	86.8
165	74.0	30	1.8	89.4	4.1	87.2
165	74.0	45	1.8	89.4	3.9	87.7
165	74.0	60	1.6	90.6	3.7	88.4
165	74.0	90	1.6	90.6	3.5	89.0
165	74.0	120	1.4	91.8	3.3	89.7
175	79.5	0	2.4	85.4	4.5	85.9
175	79.5	5	2.2	86.6	4.2	86.8
175	79.5	10	2.0	87.8	4.1	87.2
175	79.5	15	1.8	89.0	3.9	87.8
175	79.5	30	1.7	89.6	3.5	89.0
175	79.5	45	1.5	90.9	3.1	90.3
175	79.5	60	1.3	92.1	2.9	90.9
175	79.5	90	0.9	94.5	2.6	91.9
175	79.5	120	0.7	95.7	1.8	94.4
185	85.0	0	1.8	89.0	3.4	89.0
185	85.0	3	1.6	90.2	2.8	90.9
185	85.0	5	1.4	91.5	2.0	93.5
185	85.0	10	1.1	93.3	1.7	94.5
185	85.0	15	0.5	97.0	0.9	97.1
185	85.0	30	0.0	100.0	0.2	99.4
185	85.0	45	.....	.....	0.0	100.0
190	88.0	0	1.1	93.7	2.6	91.6
190	88.0	3	0.9	94.8	1.6	94.8
190	88.0	5	0.6	96.6	1.1	96.4
190	88.0	10	0.0	100.0	0.4	98.7
190	88.0	15	.....	.....	0.0	100.0
195	90.5	0	0.4	97.7	1.1	96.4
195	90.5	3	0.0	100.0	0.0	100.0
200	93.5	0	0.0	100.0	0.0	100.0

At the initial temperature of 200° F. (93.5° C.) the enzyme was completely inactivated in both the 5 and 10% pulpy juices, while at 195° F. (90.5° C.) a holding time of 3 seconds was required for each juice. The 5% pulpy grapefruit juice at 190° F. (88° C.) and 185° F. (85° C.) required a holding time of 10 and 30 seconds, respectively, for complete inactivation, while the 10% pulpy juice required 15 and 45 seconds, respectively, to inactivate the enzyme at these two temperatures. When grapefruit juices containing the two levels of pulp were held for 120 seconds at 175° F. (79.5° C.) and 165° F. (74° C.) the inactivation of the enzyme was 95.7 and 94.4 percent for the former temperature and 91.8 and 89.7 percent for the latter temperature.

The original pectinesterase in the unheated Valencia orange juice varied from 0.0172 to 0.0202 units for the 5% pulpy juice, while the juice containing 10% pulp varied in activity from 0.0331 to 0.0400 units. Both juices had a pH of 4.1. The effect of temperature and time on the inactivation of pectinesterase in Valencia orange juice showing results in (PE.u.) ml. per ° Brix and percentage inactivation is presented in Table 2. The temperatures to which both 5 and 10% pulpy orange juices were subjected in the pasteurizer ranged from 185° F. (85° C.) to 210° F. (99° C.) with holding times from 0 to 120 seconds.

An initial temperature of 210° F. (99° C.) was necessary to inactivate the enzyme completely in the Valencia orange juices containing 5 and 10% pulp. To obtain complete inactivation of the enzyme for the orange juice with 5% pulp at temperatures of 205° F. (96° C.), 200° F. (93.5° C.), 195° F. (90.5° C.), and

TABLE 2

Heat inactivation of pectinesterase in Valencia orange juice (pH 4.1)

Temperature		Holding time	5% pulp		10% pulp	
			(PE.u.) per °Brix	Inactivation	(PE.u.) per °Brix	Inactivation
°F.	°C.	*seconds*	ml. × 10^3	%	ml. × 10^3	%
185	85.0	0	1.9	89.4	2.7	91.8
185	85.0	5	1.4	92.2	2.4	92.8
185	85.0	10	1.2	93.3	2.3	93.1
185	85.0	15	1.1	93.9	2.0	94.0
185	85.0	30	0.8	96.0	1.8	94.6
185	85.0	60	0.6	97.0	1.3	96.1
185	85.0	90	0.5	97.5	1.0	97.0
185	85.0	120	0.3	98.5	0.8	97.6
190	88.0	0	1.6	91.3	2.4	92.8
190	88.0	3	1.4	92.4	2.3	93.1
190	88.0	5	1.2	93.4	1.9	94.3
190	88.0	10	1.0	94.5	1.7	94.9
190	88.0	15	0.9	95.1	1.5	95.5
190	88.0	30	0.7	96.2	1.3	96.1
190	88.0	45	0.5	97.3	1.0	97.0
190	88.0	60	0.3	98.5	0.7	97.9
190	88.0	90	0.2	99.0	0.5	98.5
190	88.0	120	0.0	100.0	0.0	100.0
195	90.5	0	1.1	93.6	2.1	94.4
195	90.5	3	1.0	94.2	1.8	95.2
195	90.5	5	0.5	97.1	1.4	96.4
195	90.5	10	0.2	98.8	1.1	97.3
195	90.5	15	0.0	100.0	1.0	97.5
195	90.5	30	.....	.....	0.3	99.2
195	90.5	45	.....	.....	0.2	99.5
195	90.5	60	.....	.....	0.0	100.0
200	93.5	0	0.7	95.9	1.4	96.3
200	93.5	3	0.3	98.3	1.2	96.8
200	93.5	5	0.0	100.0	1.0	97.3
200	93.5	10	.....	.....	0.7	98.2
200	93.5	15	.....	.....	0.3	99.2
200	93.5	30	.....	.....	0.0	100.0
205	96.0	0	0.3	98.3	0.7	98.1
205	96.0	3	0.0	100.0	0.5	98.7
205	96.0	5	.....	.....	0.3	99.2
205	96.0	10	.....	.....	0.0	100.0
210	99.0	0	0.0	100.0	0.0	100.0

190° F. (88° C.) a holding time of 3, 5, 15, and 120 seconds, respectively, was necessary. When the orange juice with 10% pulp was heated to the above temperatures, complete inactivation of the enzyme was obtained when the juices were held for 10, 30, 60, and 120 seconds, respectively. For a temperature of 185° F. (85° C.) and holding time of 120 seconds, 98.5% of the enzyme was inactivated in the 5% pulpy juice while 97.6% was inactivated in the juice containing 10% pulp.

It is readily seen from the data presented that when grapefruit juice containing 5 and 10% pulp is heated initially to 165° F. (74° C.) only 84.1 and 83.4% of the enzyme is inactivated and that when orange juice containing 5 and 10% pulp is heated initially to 185° F. (85° C.) only 89.4 and 91.8% of the enzyme is inactivated. Since these temperatures did not give complete inactivation of the pectintesterase when the juices were held for 2 minutes, lower temperatures with longer holding times would hardly seem desirable for the commercial processor.

Effect of pH upon inactivation of pectinesterase by heat in Duncan grapefruit and Valencia orange juices. Duncan grapefruit juice containing a pulp content of 10% and a natural occurring pH of 3.5 was adjusted with citric acid to pH 3.0 and with sodium hydroxide to pH 3.8. Hence, the range of pH 3.0 to 3.8 usually found in grapefruit juices was represented. The initial temperatures were varied from 135° F. (57° C.) to 205° F. (96° C.) and the juices were not held but flashed under vacuum for instant cooling. The activity and percentage of inactivation of pectinesterase in Duncan grapefruit juice at three pH levels are presented in Table 3. At pH 3.0, 3.5, and 3.8, complete inactivation was obtained with temperatures of 195° F. (90.5° C.), 200° F. (93.5° C.), and 205° F. (96° C.), respectively. As the pH was increased from 3.0 to 3.8 the percentage of inactivation was decreased for each temperature used.

TABLE 3

The effect of pH on the heat inactivation of pectinesterase in Duncan grapefruit juice containing 10% pulp

Temperature		pH 3.0		pH 3.5		pH 3.8	
		(PE.u.) per °Brix	Inactivation	(PE.u.) per °Brix	Inactivation	(PE.u.) per °Brix	Inactivation
°F.	°C.	ml. × 10^3	%	ml. × 10^3	%	ml. × 10^3	%
135	57.0	5.2	86.1	23.9	25.1	33.6	9.9
145	63.0	4.5	87.9	15.5	51.4	29.0	22.3
155	68.5	4.3	88.5	7.1	77.7	16.6	55.5
165	74.0	4.1	89.0	5.3	83.4	6.4	82.9
175	79.5	3.8	89.8	4.5	85.9	5.7	84.7
185	85.0	2.8	92.5	3.4	89.0	4.5	87.1
190	88.0	1.2	96.8	2.6	91.6	4.2	88.7
195	90.5	0.0	100.0	1.1	96.4	3.3	91.2
200	93.5	.....	.....	0.0	100.0	1.5	96.0
205	96.0	.....	.....	.....	.....	0.0	100.0

For example, in grapefruit juice heated to 195° F. (90.5° C.), with pH 3.0, 3.5, and 3.8, the percentage of inactivation decreased in the order of 100.0, 96.4, and 91.2.

A range of pH 3.2 to 4.1 is generally found in orange juice. Valencia orange juice with a pulp content of 10% and with a natural occurring high pH of 4.1 was adjusted to pH 3.6 and 3.2 with citric acid. For experimental runs with orange juice initial temperatures varied from 135° F. (57° C.) to 210° F. (99° C.) and the heated juices were immediately cooled by flashing under vacuum. Data showing the effect of pH on heat inactivation of pectinesterase in Valencia orange juice are presented in Table 4. At pH 3.2, 3.6, and 4.1 complete inactivation of the enzyme resulted at 200° F. (93.5° C.), 205° F. (96° C.), and 210° F. (99° C.), respectively. As was found in grapefruit juice the percentage of enzyme inactivated by heat was decreased with increased pH for each temperature used. Also, sharp increases were found in the percentages of enzyme inactivation for each pH as the temperatures were

TABLE 4

The effect of pH on the heat inactivation of pectinesterase in Valencia orange juice containing 10% pulp

Temperature		pH 3.2		pH 3.6		pH 4.1	
		(PE.u.) per °Brix	Inactivation	(PE.u.) per °Brix	Inactivation	(PE.u.) per °Brix	Inactivation
°F.	°C.	ml. × 10^3	%	ml. × 10^3	%	ml. × 10^3	%
135	57.0	24.5	28.2	31.7	19.5	.....	.....
145	63.0	6.7	80.4	24.0	39.1	36.8	5.9
155	68.5	2.7	92.1	9.8	74.1	33.8	13.6
165	74.0	2.4	93.0	3.8	90.4	20.7	47.1
175	79.5	2.1	93.8	2.6	93.4	8.6	78.0
185	85.0	1.3	96.2	2.3	94.1	2.7	91.8
190	88.0	0.7	98.0	2.1	94.7	2.4	92.8
195	90.5	0.3	99.1	1.5	96.2	2.1	94.4
200	93.5	0.0	100.0	0.5	98.7	1.4	96.3
205	96.0	.....	.....	0.0	100.0	0.7	98.1
210	99.0	.....	.....	.....	.....	0.0	100.0

raised to a certain level and above this level inactivation was gradual with further increased temperatures. For example, over 90% inactivation had occurred in juices at pH 3.2, 3.6, and 4.1 with temperatures of 155° F. (68.5° C.), 165° F. (74° C.), and 185° F. (85° C.), respectively, whereas the residual 10% pectinesterase was completely inactivated by heating to temperatures of 200° F. (93.5° C.) or above.

From the results presented it is evident that the pectinesterase in grapefruit and orange juices was inactivated by heat at a lower temperature when the pH was decreased. Since the pH of grapefruit juice is usually lower than that of orange juice the enzyme in grapefruit juice should be more sensitive to heat treatment. This proved to be so because lower temperatures were required to inactivate the pectinesterase present in grapefruit juice than were required for complete inactivation of the enzyme in orange juice.

SUMMARY

The pectinesterase in Duncan grapefruit juices was completely inactivated by heating in 0.8 second to temperatures of 195° F. (90.5° C.), 200° F. (93.5° C.), and 205° F. (96° C.) when the pH of the juices was 3.0, 3.5, and 3.8, respectively.

The pectinesterase in Valencia orange juices was completely inactivated by heating in 0.8 second to temperatures of 200° F. (93.5° C.), 205° F. (96° C.), and 210° F. (99° C.) when the pH of the juices was 3.2, 3.6, and 4.1, respectively.

At inactivation temperatures lower than the above the pectinesterase in grapefruit and orange juices containing 5% pulp required shorter holding times for complete inactivation than did juices containing 10% pulp. This reduction in holding time varied from 5 to 45 seconds, depending upon the temperature and pH.

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Effect of Pure Salt on the Oxidation of Bacon in Freezer Storage[a]

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Concentration of analytical grade sodium chloride (salt) had a statistically significant direct influence on the rate of oxidation of bacon sides during curing and subsequent freezer storage at 0° F. (—17.8° C.).

There is a growing practice in this country to store cured as well as uncured pork in home freezers and freezer locker plants. This practice is especially advantageous in the case of a relatively perishable product such as bacon. However, it seems to be well established that cured bacon becomes rancid much more rapidly in the freezer than uncured sides. Commercially, green sides are frozen and stored at 0° F. (—17.8° C.) and removed for curing as needed. This procedure is ideally suited to the purposes of the commercial operator, but for home use has obvious disadvantages.

The purpose of this paper is to report a phase of a systematic study of factors contributing to oxidation of bacon in freezer storage. Such knowledge should later be useful in developing practical procedures of curing and freezer storage designed to give adequate color, flavor, and stability properties for home use.

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The work reported was a chemical study of the effect of dry curing with different concentrations of analytical grade sodium chloride (salt) upon the rate of oxidation of bacon while in cure at 38° F. (3.33° C.) and during subsequent storage at 0° F. (—17.8° C.). A dry cure with salt only is not without its directly practical aspect since a recent survey by Dunker and Hankins *(5)* indicates that a high percentage of those who do their own curing use the dry cure method and, of these, a substantial proportion use only salt. Data are also presented relating to a comparison of freezer stored green cured, and cured and smoked sides, and of smoking as well as both curing and smoking after freezer storage.

Salt has been shown by a number of investigators to have an accelerating action on the oxidation of the fat of frozen pork. Lea *(13)*, White *(22)*, Dubois and Tressler *(4)*, Weisman and Ziemba *(21)*, and Watts and Peng *(20)* are among those who have contributed to the knowledge of various aspects of its action. Lea *(17)* in 1937 showed that salt promotes the activity of unsaturated fat oxidases present in pork. He indicated that he planned to determine the effect of different salt concentrations on bacon. However, so far as we are aware

the literature does not show that he followed up this line of investigation. In 1947, Watts and Peng *(20)* presented evidence that increases in salt content up to 3% produced a progressive rise in the rate of oxidation of frozen pork sausage. An effect observed in a finely comminuted and mixed product such as pork sausage might not be appreciable in the whole tissue of bacon. Greenwood, Stritar, and Kraybill *(9)* in a study of commercial dry cured bacon with 3.38, 3.80, and 4.04% salt, and 0.0078, 0.0202, and 0.0110% of nitrite, observed that rancidity development appeared to be related to the nitrite content. White, Gibbons, and Thistle *(23)* in a study of three cures and brines of different concentrations concluded that the stability of the fat is decreased by the addition of further quantities of curing salts. Salt content in their experiment was 5.3, 5.6, and 6.1% and nitrite 0.0208, 0.0257, and 0.0296%, respectively. The bacon was not smoked and was stored at 60.5° F. (15.6° C.). Very recently, Gibbons, Rose, and Hopkins *(8)* compared the effect of salt concentration at 3, 4, and 5% levels under conditions of freezer storage as low as 10° F. (—12.2° C.). Nitrite content was not more than 0.0052% in any of the samples studied. They concluded that the concentration of sodium chloride had no significant over-all effect on oxidation. A recent article *(1)* describes the new thermocure process *(12)* developed at Griffith Laboratories, Inc., Chicago, Illinois. The enhanced keeping qualities of the bacon so processed is attributed to the low salt content, which ranges from 1.50 to 1.75%. However, the bacon is cured in 16 hours or less, contains only 0.0026% nitrite, and moreover is conditioned by antioxidants before curing.

It is therefore apparent that the relative effects of different concentrations of salt and nitrite on oxidation are obscure. The results set forth in the present paper clarify to some degree the situation with respect to salt.

EXPERIMENTAL PROCEDURE

Hogs used in these experiments were fed a standard well-balanced diet and were slaughtered at a final feedlot weight of approximately 225 pounds. The work consisted of two experiments, each involving 6 hogs. In each group the individual animals were identified by letters A through F. Due to a practice at Beltsville of slaughtering in the middle of the week, time required for grading, measuring and cutting, and the intervening week end, sides were not available until the fifth day after slaughter.

The right and left sides of each hog were cut into 6 pieces of approximately equal width. The pieces of the right and left sides from the anterior to the posterior ends were numbered 2, 4, 6, 8, 10, 12 and 1, 3, 5, 7, 9, 11, respectively. In each experiment the samples were distributed in a randomized block design. (See Table 1.) Thus were obtained 12 conditions of treatment and storage in each experiment, with a sample from each hog, and an equal number of samples from right and left sides in each condition.

Samples were dry cured by spreading the salt on them as evenly as possible. They were then placed skin side down, without touching each other, in large shallow pans. The samples were cured at 38° F. (3.3° C.) for 3 weeks. Fresh or "green" controls were put in freezer storage at 0° F. (—17.8° C.) immediately. The 3-week period represents about the maximum time usually employed in dry curing, that is, 2 days per pound. All samples frozen and stored at 0° F. (—17.8° C.) were wrapped in MSAT 300 cellophane. Frozen samples were thawed for 24 hours at 38° F. (3.3° C.) before testing. Smoking was for 24 hours at 110-130° F. (43-54° C.). The salt used in curing was A. C. S. grade. This was used because it has been reported by Dunn *(6)* and Hills and Conochie *(10)* that purity of salt may be of considerable importance in meat curing.

Experiment 1, outlined in Table 2, was a comparison of oxidation taking place in green (fresh) and in cured samples stored at 0° F. (—17.8° C.). Cured samples had been treated with 2, 3, and 4% of their weight of salt.

Experiment 2, outlined in Table 3, was a comparison of oxidation occurring in freezer-stored fresh samples, cured samples, and cured and smoked samples. At the end of freezer storage, green sides were cured and smoked, and cured sides were smoked to obtain an over-all comparison. Cured samples had been treated with 4% of their weight of salt.

Each sample in both experiments was divided into inside and outside portions for chemical analysis. The outside portion was obtained by removing 4 mm. slices from each of the 4 edges and inside portions consisted of the remainder. Chemical samples were ground and stored at —50° F. (—45.6° C.) until analyzed.

Peroxides and free fatty acids were determined on inside and outside portions. Salt and protein contents were determined on inside samples. Steam-volatile carbonyls (aldehydes and ketones) were determined on 38-week, inside samples in the first experiment. Peroxides and free fatty acids were determined by the method of Rockwood, Ramsbottom, and Mehlenbacker *(19)*. Salt was assayed by the method of Kerr *(11)* and protein by an A.O.A.C. method *(2)*. Steam-volatile carbonyls of the rendered fat were determined as 2, 4 dinitrophenyl hydrazones by a method

TABLE 1
Distribution of samples

Animal	Conditions (Treatments of samples)											
	1	2	3	4	5	6	7	8	9	10	11	12
A	1	2	3	4	5	6	7	8	9	10	11	12
B	4	3	6	5	8	7	10	9	12	11	2	1
C	5	6	7	8	9	10	11	12	1	2	3	4
D	8	7	10	9	12	11	2	1	4	3	6	5
E	9	10	11	12	1	2	3	4	5	6	7	8
F	12	11	2	1	4	3	6	5	8	7	10	9

TABLE 2
Plan of Experiment 1

Conditions	1	2	3	4	5	6	7	8	9	10	11	12
Sample	Green (control)	2% salt (control)	3% salt (control)	4% salt (control)	Green	2% salt	3% salt	4% salt	Green	2% salt	3% salt	4% salt
Storage (weeks)	3	0	0	0	19	16	16	16	38	35	35	35
Temperature of storage	0° F.	—	—	—	0° F.	0° F.	0° F.	0° F.	0° F.	0° F.	0° F.	0° F.

TABLE 3
Plan of Experiment 2

Conditions	1	2	3	4	5	6	7	8	9	10	11	12
Sample	Green (control)	Green	4% salt (control)	Green	4% salt	4% salt smoked (control)	Green	4% salt	4% salt smoked	Green	4% salt	4% salt smoked
Storage (weeks)	0	3	0	38	38	0	19	16	16	38	35	35
Temperature of storage	--	0° F.	--	0° F.	0° F.	--	0° F.	0° F.	0° F.	0° F.	0° F.	0° F.
Subsequent treatment				4% salt smoked	smoked							

developed by the writer *(7)*. These data are in the form of absorbances at 355 mμ. Bacon fat rendered at 90° F. (32.2° C.) was steam distilled as it was, and after heating to 320-338° F. (160-170° C.) for 15 minutes. The latter treatment approximates temperature and time used in cooking for palatability tests.

RESULTS

Experiment 1. Average salt content of the inside portions for the 3 curing levels was 1.62, 2.87, and 4.00%. The mean protein content for the different conditions ranged from 8.11 to 9.99%, indicating that good sample distribution was obtained.

As shown in Figure 1, freezer storage decreased the rate of oxidation of the inside portions, but had slight effect on fat deterioration in the outside portions. Oxidation of the inside portions was least at the low salt level and greatest at the highest salt level. Peroxide formation in the outside portions, as might be expected, was much more rapid than in the inside portions. However, there was no clear cut distinction between outside portions from samples of 2.87 and 4.00% mean salt content. The salt content of the outside portions was not determined, but due to desiccation it was undoubtedly higher than that of corresponding inside portions.

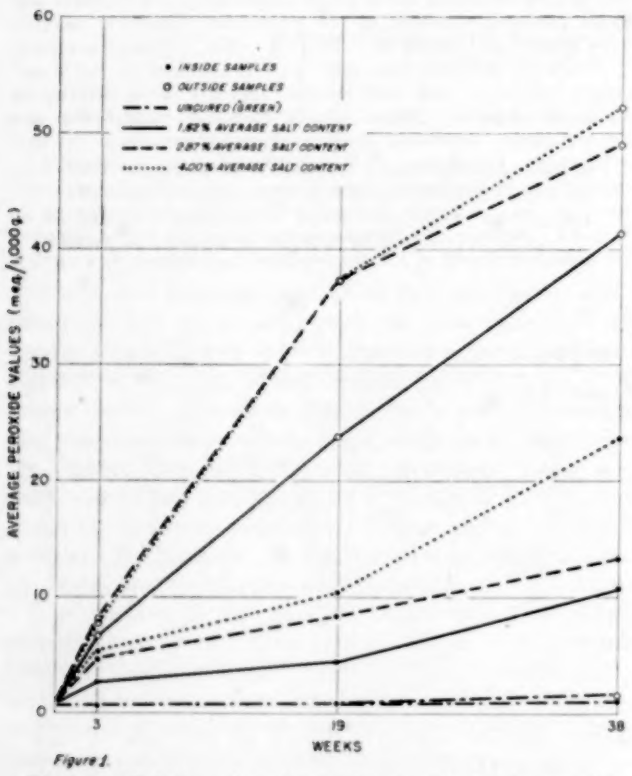

Figure 1.

Figure 1. Relation of average salt concentration to peroxide formation during three weeks of curing at 38° F. and subsequent freezer storage at 0° F. (—17.8° C.).

The relationship bewteen salt content and peroxide values at the end of the curing period, and after 16 and 35 weeks of freezer storage is shown by the regression lines in Figure 2. The correlation coefficient values

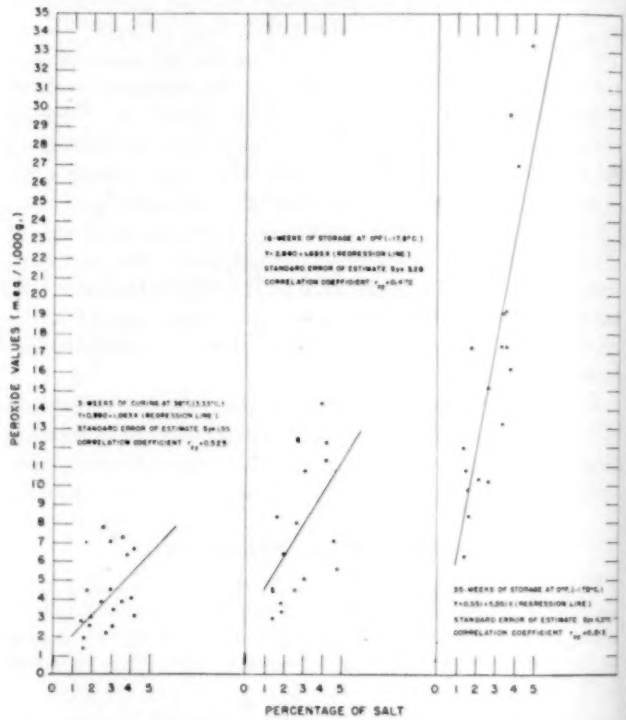

Figure 2. Relation of salt concentration to peroxide values.

shown were significant at the 5% level for the 3-weeks curing period and 16 weeks of freezer storage and highly significant at the 1% level for the 35 weeks of freezer storage. The difference between the correlation coefficients for the 3 weeks and the 16 weeks was not significant and between the 16 and the 35 weeks was highly significant. Regression lines indicate a progressive increase in rate of oxidation with increase in salt content and passage of time.

Figure 3 indicates that at 35 weeks of freezer storage the steam volatile carbonyl content of the rendered inside fat was directly related to the percentage of salt. Correlation coefficient values for unheated and heated rendered fats were +0.66 and +0.56; these values were highly significant at the 1% level and significant at the 5% level, respectively.

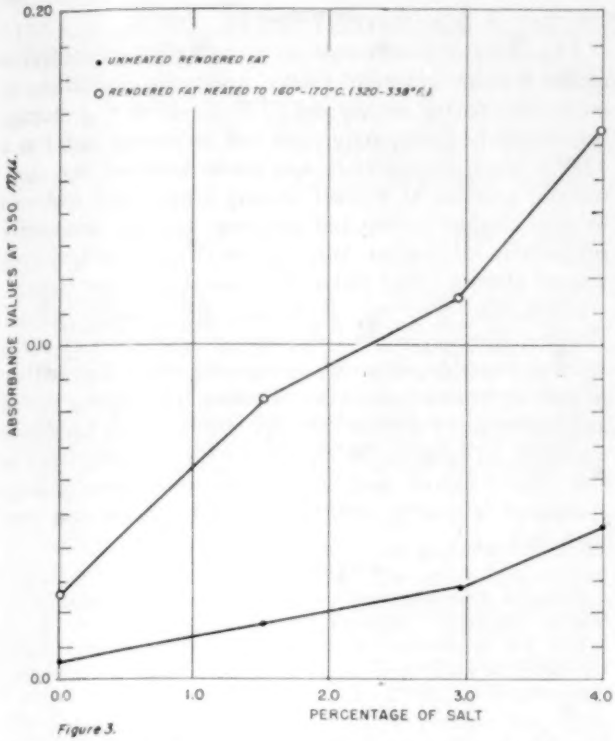

Figure 3. Relation of average salt concentration to volatile carbonyl formation (as 2,4 dinitrophenyl hydrazones) after 35 weeks of freezer storage at 0° F. (—17.8° C.).

The carbonyl compounds detected in the rendered fat were presumably those produced as the result of peroxide decomposition during storage, thawing, the mild rendering process, and during the steam distillation. The more rapid increase of the carbonyl curve of the fat heated to 320-338° F. (160-170° C.) is due to breakdown of existing peroxides not affected by previous conditions and unstable peroxides formed by the high temperature. Carbonyl compounds are believed to be responsible for rancid flavors.

Free fatty acids increased but slightly in the 2.87% and 4.00% salt outside and inside portions, and in the fresh or green samples not at all. The 1.62% salt samples had fairly substantial increases of that component in the outside portions. However, the inside portions were only slightly higher than those of the other two salt levels. Free fatty acids for the outside portions of the 1.62% samples were 3 times that of the fresh control after curing and 5 times at the end of 35 weeks of freezer storage. This suggests that microbial growth occurred on the outside during curing and this either continued during freezer storage or an enzyme was elaborated which was active at that temperature.

Experiment 2. The average salt content of the cured samples was 3.74% and of the smoked samples 4.07%. Protein content ranged from 8.63% to 10.85%.

Mean peroxide values increased to a higher level during the curing period than in similarly cured samples in the preceding experiment. The reason for this apparent difference in stability is not clear since both groups of animals were fed a similar diet. However, the comparison involved in this experiment is not adversely affected.

Reference to Figure 4 indicates, as in the previous experiment, that oxidation was slowest in the green samples and most rapid in the cured. In the freezer-stored smoked samples peroxidation took place at only a moderately greater rate than in the green. There was no significant difference in the rate of oxidation of the inside and outside portions of the green or smoked

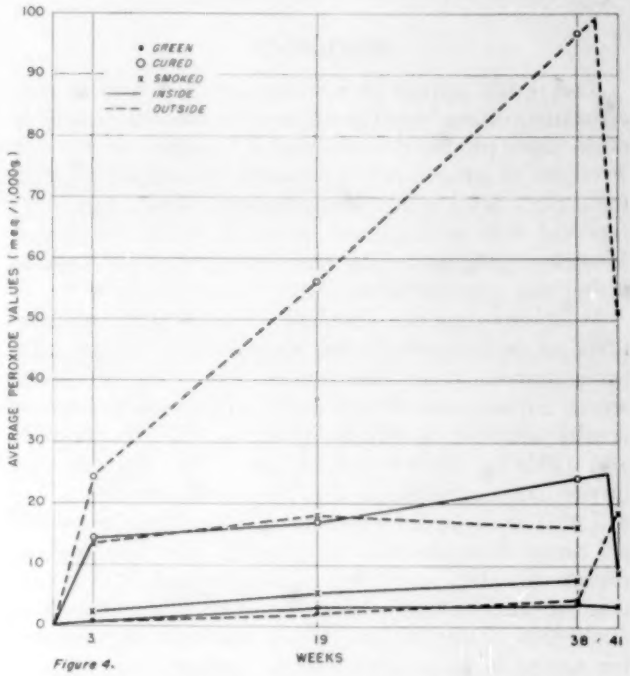

Figure 4. Relation of curing (4% salt) and smoking before or after freezer storage at 0° F. (—17.8° C.) for 35 weeks to peroxide formation.

samples during freezer storage. However, a wide difference existed between the outside and inside portions of those that were cured.

The influence of smoking upon the peroxides is interesting to note. Smoking the freshly cured samples resulted in an approximately equal decrease in peroxides in both outside and inside portions. When cured samples were smoked after 35 weeks of freezer storage the high average peroxide value of the outside portions decreased to about one-half of its previous value. The mean peroxide value of the inside portions decreased to a point which appears to nearly coincide with what the freezer-stored smoked inside portions would have been had they been examined after 3 more weeks of freezer storage. Curing and smoking the 38-week freezer-stored green samples produced outside portions with about the same apparent degree of oxidation as the outside of the freezer stored smoked samples. Peroxides of the inside portions were somewhat lower than those of the samples cured and smoked before freezer storage.

Inside portions of the three differently treated groups of bacon at the end of the experiment showed an apparent degree of oxidation not as greatly different as might have been expected. Although consideration of palatability is beyond the planned scope of the present paper, informal palatability findings indicated that some of the peroxide values may be misleading. Samples

cured and smoked after freezer storage, cured and smoked before, and cured before and smoked after freezer storage were, respectively, moderately desirable in flavor of fat for outside portions, and moderately desirable, moderately desirable, and slightly undesirable for inside portions. Thus, the bacon cured and smoked before freezer storage appeared to be nearly equal to that so processed after storage.

DISCUSSION

The results appear to indicate that increases in concentration of analytical grade sodium chloride (salt) in pork sides produced a decreased stability of the fat. Presence of active microbial growth on samples cured at the 1.62% level was a complicating factor. Lea *(14)* reported that such growth tends to inhibit oxidation. However, judging by the free fatty acids their zone of action was confined to the outside portions.

How important the observed effect of salt concentrations on oxidation is in the application of curing mixtures remains to be determined. The work of Greenwood, Stritar, and Kraybill *(9)* and consideration of results obtained by White, Gibbons, and Thistle *(23)* and Gibbons, Rose, and Hopkins *(8)* suggest that nitrite concentration is the important variable. Lea *(15, 16)*, in a model experiment, found that below pH 5.0 nitrite is a powerful pro-oxidant, and at a concentration of 0.03% oxidation is not inhibited by 5% content of antioxidant. However, Lea *(18)* expressed doubt that acidity of fatty tissue was ever high enough for nitrite to have much effect. Callow, Ingram, and Hawthorne *(3)* have shown that the pH of bacon varies from 5.50 to 5.98.

In the present work, the apparently excellent stability given by smoke ingredients to sides cured to 4% salt gives rise to some doubt whether sides cured to lower concentrations and smoked would be appreciably more stable. Most of the difference in stability might prove to be that lost during curing. Such differences could be narrowed by shortening the curing period, and perhaps removed by use of a suitable antioxidant. However, the above is mere conjecture and these points must be left to the future for clarification.

Results indicate that bacon cured with salt only and smoked will keep in freezer storage nearly as well as similar sides cured and smoked after freezer storage. Lea *(14)* stored smoked bacon, cured with a complete formula (with exception of sugar), at 14° F. (—10° C.). He found that appearance was poor and that on prolonged storage rancidity developed in the interior beyond the region penetrated by the smoke. In this connection little appears to be known regarding the extent to which smoke constituents penetrate bacon, and the chemical changes taking place. In the work reported here considerable stability appears to be conferred by smoking, not only on the outside but also on the inside portions. Moreover, the drop in peroxides produced by the smoking operation appears to be due to the action of the smoke ingredients, since the temperature [110-130° F. (43-54° C.)] of the process does not seem high enough to produce appreciable peroxide breakdown.

SUMMARY

The effect of concentrations up to 4.00% of analytical grade sodium chloride (salt) upon the oxidation of pork sides during curing and 0° F. (—17.8° C.) storage was studied. Using only pure salt as curing agent at a 4.00% level, comparison was made between the commercial practice of freezer storing fresh sides followed by post storage curing and smoking, and the alternative procedures of freezer storing cured and smoked, and freezer storing cured followed by smoking after storage.

Oxidation was rapid during the curing period. Freezer storage reduced the rate of oxidation of cured sides perceptibly, although it was still much higher than in similar frozen fresh sides. During both curing period and subsequent freezer storage the rate of oxidation appeared to be directly related to the concentration of salt. Sides cured and smoked before freezer storage compared favorably with those cured and smoked after such storage.

Acknowledgments

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The Retention of Carotene During the Canning of Tomato Juice

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Laboratory and field studies of carotene retention during the canning of tomato juice indicate that carotene is relatively unaffected by the various preparation and processing steps except for mechanical losses. The validity of calculating carotene retention on the "dry solids" basis is discussed. Data are presented showing the stability of carotene in tomato juice under aerobic and anaerobic conditions at different temperatures.

In recent years considerable attention has been directed toward investigations into the cause and extent of vitamin destruction during the preparation and heat sterilization of canned foods. In many instances losses are small enough to result in little, if any, practical decrease in the nutritional value of the food product. The reporting of significantly large decreases has quite often accompanied or preceded further investigations into the factors responsible for the loss, and many times worthwhile improvements in food processing techniques have resulted.

Along these lines carotene has generally been regarded as being fairly stable in aqueous suspensions, yet the published information on its stability during tomato juice canning *(2, 5, 8)* shows retentions ranging from 60 to 74% with an average of 67%, calculated on a dried solids basis. The loss might conceivably occur as a result of mechanical, oxidative, or thermal causes. However, several authors *(3, 4, 7)* have considered it unlikely that oxidative losses of consequence occur, except in oils, fats, or non-aqueous media. Certain plant materials, such as horse-radish, have been shown to contain highly active oxidation-promoting enzymes *(6)*, but there is no evidence reporting such substances in tomatoes. The absence of evidence of oxidative loss together with the relatively low temperatures and short processing times employed in the processing of tomato juice suggested that efforts might profitably be directed chiefly to the study of mechanical losses, such as result from screening and finishing operations. The complete study of carotene losses was thought to be quite important, since tomato juice is a good source of provitamin A. It was planned to conduct a limited but detailed survey concerning the fate of carotene during commercial canning operations, implementing such study with a preliminary set of laboratory investigations.

EXPERIMENTAL

The analytical procedure used to determine carotene was essentially that recommended by the Association of Vitamin Chemists *(1)*. An additional extraction step was adopted, resulting in recoveries of 96% or better when working with tomato skins, seeds, and cores. Total solids determinations and calculations of carotene concentrations on a dried solids basis have, in the work previously cited, provided the means for final comparisons. However, this technique does not in itself furnish an accurate measure of the amount lost during various operations, as will be amply demonstrated. In order to obtain the desired information the weight of each lot of material analyzed was obtained, thus enabling the actual weight of carotene in each fraction to be determined.

The preliminary experiment to investigate the effect of mechanical losses was conducted as follows:

A lot of quartered fresh tomatoes was divided into two portions. One portion was analyzed for carotene as representing the original material. The other was weighed, crushed thoroughly, and pressed through several layers of cheesecloth, thus separating the juice from the skins and seeds. In this operation a considerable amount of pulp was also removed resulting in a juice that was much thinner than average commercial tomato juice. These fractions were weighed and analyzed for carotene. The data collected are presented in Table 1.

TABLE 1
Cheesecloth-expressed tomato juice

	Total weight	Carotene concentration	Total carotene in fraction
	g.	*mg. per 100 g.*	*mg.*
Whole tomatoes	729	0.676	4.93
Skins, seeds, etc.	242	1.147	2.78
Expressed juice	487	0.411	2.00
Total carotene recovered			4.78 mg.
Percent carotene recovered			97.0

By converting the total weights of the fractions and their carotene concentrations into terms of total carotene, a carotene balance table was obtained which readily differentiated between mechanical and chemical losses. The 4.78 mg. or 97% recovery was indicative of the stability of carotene during the course of the experiment, and confirmed previous statments concerning the oxidative loss of carotene under operations at room temperature. However, the presence of a considerable amount of carotene in the solids fraction suggested the need for a further investigation of mechanical losses.

Accordingly, a pilot plant operation was set up, in which fresh tomatoes were chopped in a Fitzpatrick comminuter, weighed, then passed through a laboratory size "Indiana" finisher, thus simulating a cold break process. The resultant juice was weighed, as were the waste skin and seed fractions. Two finisher screen sizes were used. Additional runs were made in which the material was heated before finishing, thus simulating a hot break operation. Carotene analyses on the various fractions resulted in the data shown in Table 2.

As before, the consistently good carotene recoveries amply demonstrated the absence of oxidation. A very substantial percentage (25-30%) of the carotene was found in the refuse. These data show that the carotene concentration in the skins, seeds, and other refuse was about double of that in the finished juice and that about 15 to 17% of the original weight of tomatoes was lost as

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 11, 1952.

TABLE 2
Laboratory study of carotene retention during tomato juice extracting operations

	Weight	Carotene concentration	Total carotene	Portion of original carotene
	kg.	*mg. per 100 g.*	*mg.*	%
Cold break, screen size 0.023 inches				
Original tomatoes	6.80	0.680	46.2	(100)
Juice	5.69	0.656	37.3	80.7
Skins, seeds, etc.	1.10	1.021	11.2	24.3
Total carotene recovery				105.0%
Carotene retention				96.5%
Carotene utilization efficiency				80.7%
Cold break, screen size 0.033 inches				
Original tomatoes	7.09	0.680	48.2	.....
Juice	6.28	0.644	40.5	84.0
Skins, seeds, etc.	0.80	1.102	8.9	18.5
Total carotene recovery				102.5%
Carotene retention				94.7%
Carotene utilization efficiency				84.0%
Hot break, screen size 0.023 inches				
Original tomatoes	7.37	0.641	47.3	.....
Juice	6.26	0.584	36.5	77.2
Skins, seeds, etc.	1.12	1.056	11.8	24.9
Total carotene recovery				102.1%
Carotene retention				91.3%
Carotene utilization efficiency				77.2%
Hot break, screen size 0.033 inches				
Original tomatoes	7.40	0.641	47.4	.....
Juice	6.14	0.557	34.2	72.3
Skins, seeds, etc.	1.27	1.115	14.1	29.7
Total carotene recovery				102.0%
Carotene retention				87.0%
Carotene utilization efficiency				72.3%

refuse. It was considered likely that the small-scale pilot operation resulted in far more residual material than would ordinarily be encountered in commercial production. It will be noted from the carotene retentions shown in Table 2 that the series of results calculated on the basis of the carotene concentration of the original tomatoes and finished juice did not agree with the carotene utilization efficiencies, which term has been defined as the extent to which the carotene present in the original raw tomatoes was utilized in the finished juice.

The experiment to determine the extent of carotene losses at processing temperatures was conducted as follows:

A container was half-filled with tomato juice, then the juice and the headspace were gassed with either air or nitrogen. The container was then sealed and given thermal treatment while being agitated continually so as to maintain gas saturation in the juice. Carotene concentrations were determined initially and after 1 hour of treating time. The data collected are shown in Table 3.

TABLE 3
Carotene decomposition rate at various processing temperatures

Temperature	Carotene lost per hour	
	Nitrogen-saturated	Air-saturated
° F.	%	%
180	0.0	0.0
205	2.5	4.0
240	0.0	9.1
260	15.3	37.8

From these results it was expected that small but measurable losses in carotene content could occur during flash sterilization or boiling water cooks.

With the above experiments as a background the commercial operations of two Indiana tomato juice canneries were investigated.

The first plant used a hot break followed by a shaker screen, extractor, and tubular sterilizer. The carotene survey covered a full 5-hour operational period. Careful checks were made of the amounts of material entering the plant, the waste, and the resulting canned juice. Losses resulting from trimming and spillage were negligible in comparison to the total amount of material handled. Samples were taken at various points along the canning line at half-hourly intervals. The carotene values thus obtained for each station were averaged, the average figure being taken as representative of that station during the entire period.

TABLE 4
Study of carotene retention at cannery No. 1
Hot break presterilized juice

	Total weight	Carotene concentration	Total carotene	Solids [b]	Carotene in solids
	tons	*mg. per 100 g.*	*g.*	%	*mg. per 100 g.*
Original chopped tomatoes	43.2	0.591	233	5.7	10.5
Shaker pulp	1.2	1.370	14	11.9	11.5
Extractor pulp	1.3	1.147	15	21.4	5.4
Finished juice	40.7	0.562	207	5.0	11.1
Totals	43.2		236		
Juice after canning		0.522	192	5.0	10.4
Over-all retention					88.3%
Over-all retention on solids basis					99.0%
Carotene utilization efficiency					82.5%

[b] Salt-free solids.

Table 4 summarizes the data collected. Out of 43.2 tons of material, only 2.5 tons were discarded from the shaker and the extractor. As in previous experiments, the carotene concentration in the shaker and the extractor pulp was about double of that in the finished juice. However, the small weight of material thus discarded limited the carotene lost to 29 g. out of an original 233 or 12.5%. The total carotene recovery of 236 g. compared with the 233 g. known to be present in the original material indicated that all of the carotene was present in the finished juice and discarded portions. The carotene utilization efficiency of 82.5% on the total carotene basis was a measure of the degree to which the tomato carotene entering the cannery was utilized in the final product. The adjustment of juice extractors so as to result in higher juice yields also resulted in a higher concentration of carotene in the final juice. This offers a practical means for increasing the carotene content of tomato juice.

The second plant used hot filling together with air cooling. The carotene survey was conducted as described above, except that smaller lots of material were used. The data collected is summarized in Table 5 and includes both hot break and cold break operations. It will be observed that there is considerably lower carotene utilization efficiency in Cannery No. 2 as compared with Cannery No. 1. The similarly lower over-all retentions of carotene on the basis of the "as received" samples can be explained by the fact that in Cannery No. 2 for the two operations stated, the extractor and finished refuse amounted to 18.3 and 24.2%.

DISCUSSION

A common method of expressing vitamin retentions during canning operations is on a dried solids basis,

TABLE 5
Study of carotene retention at cannery No. 2

	Total weight	Carotene concentration	Total carotene	Solids[c]	Carotene in solids
Hot break	*tons*	*mg. per 100 g.*	*g.*	*%*	*mg. per 100 g.*
Original chopped tomatoes	0.82	0.592	4.38	5.70	10.38
Skins, seeds, etc.	0.15	1.067	1.40	9.01	11.85
Finished juice	0.67	0.511	3.11	4.99	10.24
Totals	0.82		4.51		
Juice after canning		0.482	2.94	5.00	9.64
Over-all retention					81.4%
Over-all retention on solids basis					92.9%
Carotene utilization efficiency					71.0%
Cold break					
Original chopped tomatoes	0.82	0.564	4.17	5.88	9.59
Skins, seeds, etc.	0.20	0.702	1.26	7.36	9.54
Finished juice	0.62	0.482	2.70	4.92	9.80
Totals	0.82		3.96		
Juice after canning		0.462	2.59	5.00	9.24
Over-all retention					81.9%
Over-all retention on solids basis					96.4%
Carotene utilization efficiency					62.2%

[c] Salt-free solids.

which gives misleading interpretations so far as over-all retentions are concerned. For example, in Table 4, the 99% value was calculated on a salt-free solids basis, and reflects nothing more than the ratio of the carotene compositions of the original and final solids. The solids content of the final juice, if 0.4% added salt were included, would be 5.4% instead of 5% and the retention of carotene on a dried solids basis would then be only 92.2%.

A review of Tables 4 and 5 reveals that over-all carotene retentions have been calculated on two different bases, namely from carotene contents expressed in terms of "as received" samples, and of the dry solids in the samples. As a third basis, carotene utilization efficiency is a measure of total carotene retention in the cannery and is calculated from the total weight of carotene in the original tomatoes, and in the final canned juice. A consideration of the methods used for calculating these values will reveal that the retentions from the three sets of figures for each lot have three different meanings and should not be compared with one another.

From the nutritional standpoint, the consumer is naturally interested in obtaining tomato juice as closely approximating the composition of the original tomatoes from which the juice was prepared as is possible. Retentions of carotene calculated on the fresh basis rather than the dried solids basis are, in the opinion of the authors, the best values to indicate the relationship of carotene content of the final product to that of the natural material from which the product has been prepared. Such values on a percentage basis express a comparison of the canned product to the composition of the source material, but do not necessarily include all the changes which may take place during the canning operations.

By calculating the total carotene content of the original tomatoes, the various waste portions, and the final juice after canning, one obtains a balance sheet from which the utilization efficiency of carotene in the plant may be readily apparent. This value should not necessarily be in agreement with the over-all carotene retentions for the finished canned product, as calculated from the carotene content of the "as received" samples, or with the "over-all carotene retentions" calculated on the basis of the dried solids. The carotene utilization efficiency is a measure of the cannery's efficiency in retaining the total carotene in the tomatoes which have entered the plant in the final canned product, but does not necessarily present the relationship of the carotene composition of the final canned product to that of the original raw tomatoes from which the juice was prepared.

As has been mentioned, data previously reported on carotene retentions during the canning of tomato juice *(5)* have been mainly derived from a comparison of the carotene content of the dry solids from the juice after canning with that of the original chopped tomatoes. When the nutrient under consideration is an integral part of the solids, this makes the situation more complex than when the nutrient is soluble in the liquid and not fixed in its entirety with the solids, particularly in evaluating a process where some of the preparatory operations are specifically designed to take out solid materials.

SUMMARY

The investigative techniques employed here have demonstrated that carotene retentions during the canning of tomato juice need not be considered as low as those previously published in the literature. The "as received" basis has appeared to be of the most value in correctly expressing the nutritional quality of the juice as reflected by the canning operations. Calculated in this manner, retentions from the surveys discussed in this report were 81.4% and 88.3% for two hot break operations and 81.9% for one cold break operation. Carotene losses before processing were shown to be entirely the result of mechanical separation. Thermal processing caused small, but measurable losses. However, the over-all results indicate that carotene may be regarded as a relatively stable substance in ordinary canning operations.

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Antioxidants and Food Preservation[a]

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Efforts to retard oxidative rancidity in fats and fat-containing foods frequently fail because of an incomplete understanding of antioxidants and their use. General suggestions are presented on the selection, concentration and application of antioxidants to a variety of foods. Data illustrate the increased storage life which can be expected.

Chemicals have long been used in the food industry to delay bacteriological and enzymatic decomposition. Within the last few years, food processors are realizing the value of antioxidants to retard development of the unpleasant flavor, odor, and color characteristics of oxidative rancidity. These undesirable properties are the result of the action of fats and oils contained in fatty foods with the oxygen in the air to form products such as aldehydes and acids which in turn are directly responsible for rancidity. Flavor, odor, and color often can be preserved by the proper use of the right antioxidant. Such chemicals as butylated hydroxyanisole (BHA), propyl gallate, nordihydroguaiaretic acid (NDGA), and citric acid are readily available at prices which permit the stabilization of fats and fatty foods at very low cost.

Butylated hydroxyanisole is by no means a new product to the food industry. It has been widely publicized in scientific journals *(3, 4, 5, 6, 7, 8, 9, 10)*. Most of the work, however, has been on the stabilization of animal fats(lard, oleo oil, beef tallow) and vegetable oils (cottonseed, hydrogenated cottonseed, peanut, and corn oil) and their applications to baking and deep fat frying. This article is directed toward the use of antioxidants as preservatives for the fatty components of foods.

SELECTION OF THE PROPER ANTIOXIDANT

As an aid in the selection of the best antioxidant for a specific application, some of the outstanding properties of the more common food grade antioxidants are presented.

Butylated hydroxyanisole (BHA) is a combination of the 2- and 3-isomers of monotertiarybutyl-4-hydroxyanisole. It is readily soluble in practically all fats and oils and insoluble in water. It is stable to heat and is not destroyed or lost during cooking, frying, and baking processes; therefore, BHA extends the storage life of baked and fried foods. Since it is stable to mild alkali, BHA is particularly suited to the baking industry where alkaline conditions are often encountered. Butylated hydroxyanisole is not discolored by contaminating metals such as iron.

Propyl gallate is the normal propyl ester of gallic acid. It has a limited solubility in fats and oils, less than 2.0%, and is rather difficult to dissolve in fats and fatty foods without the aid of a carrier solvent. It is quite soluble in water, the degree of solubility increasing with the temperature of the solution. Although propyl gallate provides excellent storage life to neutral or slightly acid fats and fatty foods which are not subjected to elevated temperatures, it is unstable to heat and mild alkali. Like most polyhydroxy aromatic compounds it discolors in the presence of iron. The blue-black colored reaction product of propyl gallate and iron can be minimized and often eliminated if the propyl gallate is properly handled and a powerful metal deactivator such as citric acid is employed. Stainless steel equipment is recommended wherever propyl gallate is used.

Nordihydroguaiaretic acid (NDGA) is extracted from the desert plant *Larrea divaricata*, one of several species of plants commonly known as creosote bush. As an extract, the cost of the compound is relatively high. It is only sparingly soluble in fats, approximately 0.5%, and is rather difficult to dissolve without the aid of a carrier solvent. It is practically insoluble in water. Like propyl gallate, NDGA provides excellent storage life to neutral or slightly acid fatty foods which are not subjected to elevated temperatures; it is unstable to high temperatures and mild alkali. NDGA does not discolor in the presence of iron and is recommended where propyl gallate is unsatisfactory.

Citric acid has long been known as an excellent sequestering agent, effectively complexing contaminating pro-oxidative metals such as iron and copper. It is only slightly soluble in fats, less than 0.005%, and quite soluble in water. With the aid of a carrier solvent such as propylene glycol, citric acid can be easily dissolved at greater than sufficient concentrations to sequester the small amounts of contaminating metals picked up during the processing of fats or fatty foods.

Propylene glycol is a unique solvent for propyl gallate, NDGA, citric acid, and synergistic combinations of these with the readily oil soluble BHA. The propylene glycol permits easy solubility of all components within limits considerably in excess of the small amounts required for the stabilization of fats.

Each of the described chemicals has certain properties which adapt it to specific applications. These properties should always be kept in mind when selecting an antioxidant. Frequently a synergistic combination of antioxidants proves more desirable than any one antioxidant. Many such synergistic combinations are commercially available.

CONCENTRATION OF THE ANTIOXIDANT

Generally speaking, stabilization of fats and oils can be accomplished with 0.01% BHA, propyl gallate, or NDGA. If the fats or oils are to be subjected to continuous use such as heavy duty frying, 0.02% BHA is recommended. The Meat Inspection Division of the Bureau of Animal Industry approves the use of BHA at 0.02%, propyl gallate and NDGA at 0.01%, and citric acid at 0.005% concentrations when they are used either alone or in synergistic combination.

There is some difference of opinion as to whether the antioxidant concentration should be based upon the oil content or the total weight of the food. The ratio of fat to other components in the food and the method of stabilization should be considered. If there is a high percentage of protein, carbohydrate, or other material and a low percentage of fat, the required antioxidant concentration should be calculated as a proportional

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 11, 1952.

percentage of the total weight of the food product. The nonfatty components may absorb much of the antioxidant, depriving the fat of protection. If the antioxidant is applied to the surface of a food and expected to remain there, reduced concentrations eliminate the possibility of detecting any flavor when the food is consumed.

APPLICATION OF THE ANTIOXIDANT

The method of applying or incorporating the antioxidant into the food determines, to a large extent, one's success or failure. The antioxidant must be dissolved in the fat or fatty component of the food to be protected. Although the stabilization of every food product is an individual challenge, variations of one or more of the following methods of application should prove successful:

1, solution; 2, addition; 3, painting and dipping; 4, spraying; and 5, injection and pumping.

Solution. Solution is dissolving the antioxidant in a fat or oil which is later added to the food. Generally, this is the simplest and most satisfactory method. It can be used for the stabilization of animal fats, vegetable oils and any foods to which these fats and oils are added. This method has proven satisfactory for cream, butter, candy, pastry premix, citrus oils, essential oils, vitamin A in natural fish oils, oleoresin capsicum, chicken fat, oleomargerine, cosmetics, and paraffin wax.

BHA can be dissolved easily in fats and oils by merely stirring the BHA-liquid fat combination sufficiently to insure uniform distribution of the BHA throughout the solution. Synergistic combinations of BHA with propyl gallate, NDGA, or citric acid require a carrier solvent and good agitation for about 25 minutes at 140-176° F. (60-80° C.) to insure uniform distribution and true solution of the less soluble components.

Addition. Addition is adding the antioxidant directly to the food product without first dissolving it in some fatty component. This method has proven successful with corn and wheat germ meal, butter, chewing gum, peanut butter, and dog food. It is important to have adequate mixing facilities; that is, equipment which will provide a uniform distribution of the antioxidant throughout the entire mass of the product to be stabilized. Dilution of the antioxidant with a suitable solvent facilitates distribution.

BHA is particularly applicable to the direct addition method because of its ready solubility in oil. Propyl gallate and NDGA require a carrier solvent. In such applications, a synergistic combination of antioxidants is often desirable.

Painting and dipping. Painting and dipping offer alternate procedures for stabilizing those foods which do not lend themselves to the previously described methods. They provide a convenient means of surface application and have been successfully used to retard the oxidation of bacon, ham, beef, and frozen fish and to treat food grade wrapping papers.

Again, BHA is especially suited to this method of application because of its ready solubility in the fatty components of the food and its insolubility in any water which may be present. BHA can be diluted to a suitable concentration with oil. A combination antioxidant can be diluted with a food grade organic solvent or an aqueous emulsion can be prepared. Propyl gallate can be dissolved in water.

Spraying. Spraying offers another convenient way of stabilizing fat-containing foods by the surface application of an antioxidant. A spray technique has been successfully applied to dog food, cereals, ground meal, nuts (peanuts, walnuts, pecans), shredded coconut, fish, meat, and paper and paperboard used for packaging fat-containing foods.

Spraying uniformly distributes the antioxidant and reduces or eliminates, depending upon the product, the necessity of mechanically mixing the antioxidant throughout the food. Most products can be sprayed with as little as 0.2-2.0% aqueous emulsion. It is sometimes necessary to remove the excess water by passing the stabilized product through a drier.

Injection and pumping. Injection and pumping enable the stabilization of many other foods. By these methods, the antioxidant can be brought directly in contact with the fatty interior of foods, without the time lag required for the diffusion of the antioxidant from the exterior. Injection and pumping have been satisfactorily used with bacon, ham and fish. An aqueous emulsion of the antioxidant is recommended.

Aqueous emulsions of BHA and its synergistic combinations can be prepared with gum tragacanth as the emulsifier and propylene glycol as an introfier. A typical emulsion consists of 50 parts of Tenox II,[b] 5 parts of gum tragacanth, and 45 parts of water. The gum tragacanth should be dispersed in the water and allowed to swell for 12 hours to form a smooth, viscous paste. The Tenox II should be slowly stirred into the paste. The resulting emulsion can be diluted to the required antioxidant concentration or viscosity by the careful addition of more water. Each increment of water should be completely assimilated by the emulsion before further additions are made.

EXPERIMENTAL DATA

Some conception of the increased storage life of fatty foods which can be obtained by treating them with antioxidants is presented in Table 1. Although most of these foods have been tested with BHA, propyl gallate, NDGA, citric acid, and combinations of these, for the sake of brevity, only the data on the combination antioxidant Tenox II is presented as an example.

SUMMARY

Laboratory and commercial scale experiments have proved the utility of antioxidants in fats and fat-containing foods. Tests included animal and vegetable fats and oils, fried and baked foods, dairy products, meat products, fish, nuts, cereals, condiments, vitamin A, and food grade paper and paperboard.

Butylated hydroxyanisole prolonged the storage life of uncooked foods, extended the frying life of fats and oils and "carried-through" baking and frying processes

[b] Tenox II contains 20% butylated hydroxyanisole, 6% propyl gallate, 4% citric acid, and 70% propylene glycol. Manufactured by Tennessee Eastman Company, Kingsport, Tennessee.

TABLE 1
Percent stability[c] increase with Tenox II

Food tested	Tenox II added	Stability (days) Schaal oven life 145° F. (63° C.) Unstabilized	Stabilized	Stability increase
	%			%
Potato chips[d]	0.05[e]	1	33	3,200
Crackers[d]	0.05	7	25	257
Pastry[d]	0.05	3	24	700
Butter	0.05	15	54	260
Wheat germ meal	0.05[f]	6	54	800
Corn meal	0.05	36	94	161
Pecans	0.05	28	40	43
Walnuts	0.05	35	55	57
Lard film on:				
Wax paper	0.05[g]	4	7	75
Paperboard	0.10	1	5	400
Pastry premix in:				
Parchment paper	0.10	5	14	180
		Refrigerator storage at 34-36° F. (1-2° C.)		
		Unstabilized	Stabilized	
34% cream	0.10[e]	8	16	100

[c] The number of days before oxidative rancidity can be detected organoleptically.
[d] Lard used as the frying and baking fat.
[e] Percents based on oil content of food.
[f] Percents based on total weight of food.
[g] Percents based on total weight of wrapping paper.

to increase the storage life of cooked foods. In other experiments, this antioxidant preserved the natural color of oleoresin capsicum and tuna flesh. Protection against oxidative rancidity was afforded to packaged fats and fatty foods by treating parchment and glassine paper, paraffin wax and wax-coated paper and paperboard with butylated hydroxyanisole.

The addition of propyl gallate, nordihydroguaiaretic acid, and citric acid often increased the effectiveness of butylated hydroxyanisole as an antioxidant. When properly used, neither the antioxidants nor their synergistic combination imparted any detectable flavor, odor, or color to the fat or the food.

The Pure Food and Drug Administration has sanctioned the use of these chemicals in the recommended concentrations. The Bureau of Animal Industry has granted permission for their use in Memorandum No. 118.

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A Preliminary Study of Factors Affecting the Quality of Pickles on the Canadian Market[a,b]

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Results of quality studies on pickles on the Canadian market are reported and the factors affecting quality are discussed. Studies made on the curing of cucumbers are discussed with special emphasis on the effect of brine concentration on the quality, the control of film-forming yeasts, and the methods of initiating a green color in cured cucumber stock.

The pickle industry in Canada has developed rapidly. In 1929, 1,500,000 gallons were produced; in 1950, 5,500,000 gallons. This could be further increased if the quality of pickles was uniformly high. With this in mind, the Canada Department of Agriculture initiated a study of factors affecting the quality of pickles.

QUALITY STUDIES ON PICKLES ON THE CANADIAN MARKET

As a starting point on the study of pickles microbiological and chemical analyses were made of different types and brands of pickles packed in Canada. In all, 57 samples comprising sweet, sour, and dill pickles were

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obtained from different regions. In addition to the above-noted analyses, a taste panel of 7 persons was used as a means of judging consumer acceptability. If three or more panel members criticized a sample for a specific reason, attempts were made to determine any possible connection between this fault and chemical and microbiological findings.

On the basis of chemical, microbiological, and taste panel tests, only 23 of the 57 samples (40%) were considered acceptable products. Table 1 shows that 56% of the dills, 82% of the sour mixed, 55% of the sweet mixed, and 50% of the miscellaneous samples were below the desired quality. Regionally there was considerable difference in the quality of pickles with Ontario and the Western Provinces showing the highest quality and Quebec pickles the lowest. Too few samples were examined from the Maritimes to form any definite conclusions.

TABLE 1

Results of Survey of Canadian Pickles

Region	No. of samples from region	Types of pickles	No. of samples of each type	Average: Poor quality samples	Average: Poor quality, all types
				%	%
Ontario	28	Dill	4	50	43
		Sour mixed	5	60	
		Sweet mixed	14	43	
		Miscellaneous	5	20	
Quebec	17	Dill	2	100	88
		Sour mixed	5	100	
		Sweet mixed	8	75	
		Miscellaneous	2	100	
Western Provinces	9	Dill	3	33	44
		Sour mixed	0	0	
		Sweet mixed	5	40	
		Miscellaneous	1	100	
Maritimes	3	Dill	0	0	100
		Sour mixed	1	100	
		Sweet mixed	2	100	
		Miscellaneous	0	0	
Combined Areas	57	Dill	9	56	60
		Sour mixed	11	82	
		Sweet mixed	29	55	
		Miscellaneous	8	50	

From a bacteriological standpoint, only 3 samples (2 dill and 1 sour mixed) were seriously contaminated. The dill pickles showed plate counts of 14 and 15 million, respectively, and the sour mixed pickles nearly 14 million organisms per milliliter of liquor. The salt concentrations of these dill pickles (2.5 and 2.34%, respectively) were far below those necessary to prevent the development of undesirable organisms such as pectin and cellulose decomposers. Although small numbers of organisms were found in 8 other samples, these were probably too few to have any deleterious effect.

The quality of pickles is governed largely by a proper balancing of the major chemicals used, namely salt, sugar, and acid. Although the salt concentration is of major importance in dill pickles, it is considered of lesser importance in sweet and sour pickles. Fabian and Switzer *(1)* have recommended desirable concentrations for salt, sugar, and acid and these have been used as a basis for comparing the results of analysis of Canadian pickles in Table 2. From the results presented in this table it is significant that all 3 putrid samples were low in either salt or acid concentration. The 3 samples criticized solely for tough rinds were balanced and acceptable in every other respect. None of the 24 samples of "off-flavored" pickles had the desired "balance"; 9 samples and possibly 3 others of the 24 accepted as satisfactory by the taste panel were properly balanced.

Sediment and filth analysis of the samples of pickles shown in Table 3 indicated that the majority of packers produced a clean product. There did not appear to be any correlation between the amount of sediment and filth and the taste panel findings or appearance of the pickle. Of the 57 samples, 4 showed an abnormally large amount of hairs and insect fragments.

TABLE 3

Sediment and filth [d] analysis

Classification	Number of samples	Appearance of product	Number off-flavor	Per cent off-flavor
No foreign material found	35	Very clean	20	57
Minute amount of filth	15	Clean to very clean	6	40
Moderate amount of filth	4	Dirty	3	75
Decomposed samples	3	Clean	3 (putrid)	100
Total	57	.....	32	56

[d] Insect fragments and rodent hairs.

STUDIES ON THE CURING OF CUCUMBERS

The survey of commercial pickles on the Canadian market led to the conclusion that the proper balance of various ingredients such as salt, acid, and sugar will not alone ensure that the finished pickle will be acceptable. Other factors probably include the quality of the initial cucumber stock, the variety of cucumber, and perhaps of greatest importance, the curing and handling of the salt stock. It is well known that the concentration of salt used in curing must inhibit undesirable micro-

TABLE 2

Samples showing deviations from recommended concentrations of salt, sugar and acid

Criticism	Total No.	Balanced [c] Pickles	Salt high	Salt low	Acid high	Acid low	Salt low, sugar low	Salt low, acid high	Salt high, acid high	Acid high, sugar high
Putrid	3	0	0	2	0	1	0	0	0	0
Off-flavor	24	0	6	7	5	1	2	2	1	0
Tough rind	3	3	0	0	0	0	0	0	0	0
Satisfactory	24	9	2	5	4	0	0	3	0	1

[c] Based on findings of Fabian and Switzer *(1)*.

organisms responsible for spoilage without retarding the development of acid-producing bacteria necessary for fermentation. Unfortunately, molds and yeasts, especially the film-forming yeasts, also tolerate the brine concentrations usually employed. These yeasts are important in pickle curing since they utilize the acid produced by desirable bacteria and thus produce conditions favorable for the development of spoilage organisms.

Methods

In the two seasons the curing process has been studied at these laboratories two concentrations of salt, 10% and 4%, have been employed. The original concentration of each brine was restored daily until an equilibrium between the salt concentration in the cucumbers and the brine was reached. Analyses of the brine were made daily until equilibrium was reached and then at semi-weekly and finally at weekly intervals until the 45th day. This varied somewhat according to the findings. Salt concentrations were determined by both salometer readings and titration. Other chemical determinations included soluble solids, titratable acidity, and pH.

Microorganism counts were determined using tryptone glucose extract agar both with and without salt, and nutritive caseinate agar to differentiate between acid, non-acid, and peptonizing types of bacteria.

Results

Since the data on the microbiological and chemical findings are voluminous, the results are presented in graph form in Figures 1 and 2. The growth of bacteria in the 10% and 4% brines is shown in Figure 1. When a 10% brine was used in the curing process there was an immediate reduction in the total number of microorganisms. This was followed by a levelling-off and the counts remained more or less constant until the end of the period of observation. The 10% brine series plated on tryptone glucose extract agar plus 10% salt (salt tolerant organisms) showed an immediate drop in count followed by a period of fluctuating counts to the 10th day. From the 10th day on a gradual increase was observed.

In the early period of curing, micrococci were predominant with smaller numbers of flavobacteria. However, about the 11th day yeasts and lactobacilli began to increase and about the 14th day were more numerous than other types.

The use of 4% brine for curing presented an entirely different picture. The total count as shown in Figure 1 shows a tremendous increase in numbers during the first 2 days of curing followed by a more or less stationary count throughout the period of observation. The tryptone glucose extract agar with 4% salt added to simulate the concentration in the pickle liquor also showed a significant increase in numbers of microorganisms during the first 2 days. This was followed by a steady decrease until the 6th day and then an irregular increase until the end of the observation period. It is of interest to note that throughout the curing period the total counts in the 4% brine were consistently higher than in the 10% curing brine.

While detailed studies of types of organisms were not made throughout the period of curing with the 4% brine it was noted that spore-forming bacteria were numerous particularly during the early period of curing.

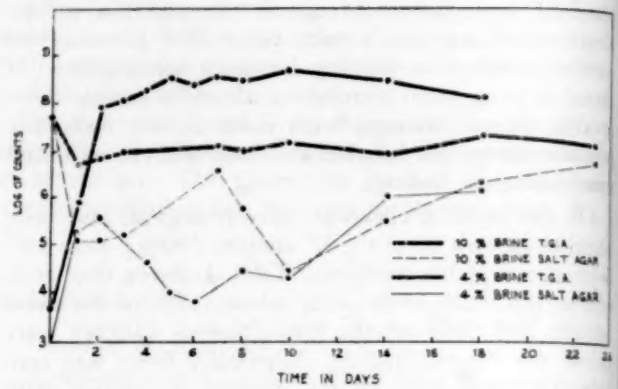

Figure 1. Growth of bacteria in two different concentrations of brine stock.

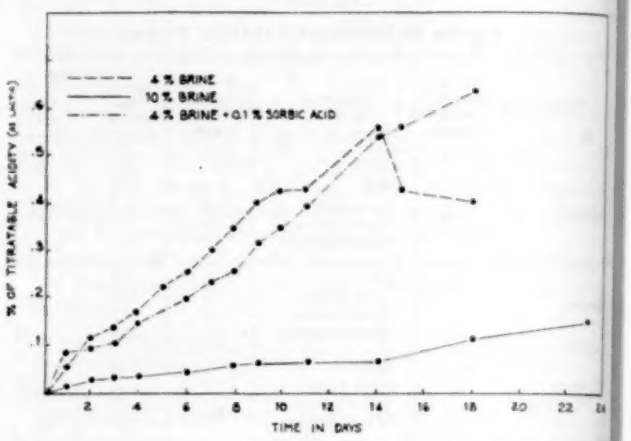

Figure 2. Acidity development in brined cucumbers.

Concurrently with the studies on microorganisms, chemical tests were conducted. The data in Figure 2 show that the total acidity produced in the 10% brine series was very low, slightly over 0.1%. Use of 4% brine for curing resulted in a maximum production of over 0.6% acid, particularly when 0.1% sorbic acid was used to control the growth of film-forming yeasts. The acidity in the 4% brine with and without 0.1% sorbic acid ran almost parallel up to the 14th day. Subsequently, the lots containing sorbic acid continued to produce acid while the 4% brine alone showed a marked reduction in the amount of acid. This was due to the utilization of the acid by film-forming yeasts which developed at this point.

It was concluded that 10% brine was inhibitory to the development of the desirable acid-producing bacteria. In addition, the removal of water from the cucumbers was too rapid at this concentration as evidenced by the visible shrivelling which occurred. The 4% brine, on the other hand, proved more favorable for these acid-producing bacteria but some difficulty was encountered with spore-formers during the early stages of fermentation.

It is of interest to note the difference in the results of salt concentration as determined by salometer reading and by titration presented in Figure 3. In every instance

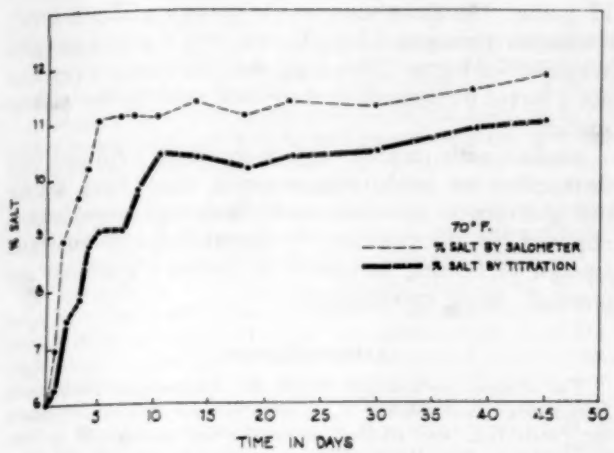

Figure 3. Comparison of salt content as determined by salometer reading and by titration.

the salometer reading was higher than that determined by titration. This was likely due to the soluble solids diffusing from the cucumber during the curing process. While these differences would not be considered important in the higher concentrations they are important in the critical salt range necessary to control the development of undesirable microorganisms.

Control of film-forming yeasts. The control of acid-consuming, film-forming yeasts presents a serious problem in the curing and storing of cucumbers. Neither the 4% nor the 10% brine had any inhibitory effect on the development of these organisms. As a point of interest, approximately 22% of salt is required to completely inhibit the development of these yeasts. Removal of the heavy surface layer of yeasts followed by ultraviolet irradiation for 2 hours daily prevented further surface growth. However, the rays did not penetrate the brine to any appreciable depth. Many of these yeasts develop below the surface of the curing brine beyond the effective range of these rays and have been found to cause undesirable changes in the cucumber stock.

The possibility of using antibiotics as a means of controlling yeasts has been studied. Results in Table 4 show that actidione was by far the most potent antibiotic but its toxicity would probably preclude its use in food products. Other anti-fungal antibiotics such as gliotoxin and citrinin showed some inhibitory power as did also the anti-bacterial antibiotic tyrothricin.

Detailed studies during the past season have been made with sorbic acid, a fungal-inhibiting chemical recommended by Phillips and Mundt *(2)*. Results of plate counts (Figure 4) indicate that there is a definite inhibition after 48 hours in the 0.05% and greater concentrations. This was also evident by visual examination of the pickle liquor which showed no evidence of surface scum in concentrations of 0.05% or greater of sorbic acid. Complete inhibition was noted at a concentration of 0.1% after 96 hours. Microscopical examination of the curing liquor showed that the sorbic acid apparently had no important effect on the development of rod-type bacteria. However, plate counts made during the period of fermentation showed that sorbic acid had a slight retarding effect on acid-forming bacteria

TABLE 4

Influence of antibiotics on film-forming yeasts

Antibiotic[f]	Inhibition[e] at 24 hours		
	Complete	Partial	None
Actidione	520.0	4160.0	8320.0
Gliotoxin	8.0		16.0
Citrinin	4.0	8.0	16.0
Tyrothricin	4.0	8.0	16.0
Aspergillic acid	2.0		4.0
Chloromycetin	2.0		4.0
Aureomycin	1.0	2.0	4.0
Streptothricin	1.0	4.0	8.0
Subtilin	1.0	8.0	16.0
Clauacin	<1.0	1.0	2.0
Streptomycin	<1.0	2.0	4.0
Bacitracin	<1.0		1.0

[e] Reciprocal of dilutions in ten-thousands giving inhibition.

[f] (EDITOR'S NOTE: The physiological effects of various antibiotics, if used in foods, has not been clearly established. Practical use of these agents in foods is not permitted at the present time.)

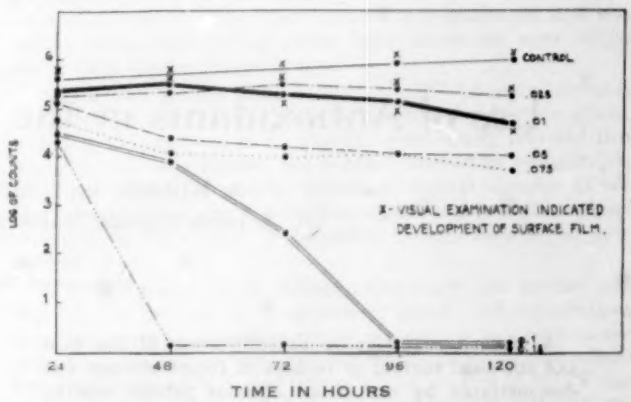

Figure 4. Effect of different concentrations of sorbic acid on film-forming yeasts in pickle brine.

in the early stages of curing. As noted previously (Figure 2) following the 14th day the acidity of those brines containing 0.1% sorbic acid continued to increase while the untreated brines showed an appreciable drop in acidity due to the development of large numbers of acid-consuming yeasts.

Color studies on freshened cucumber salt stock. While there are some consumers who consider color secondary in evaluating quality, it has been our experience that most people prefer a green pickle.

The color of cucumbers in the stage of harvesting for pickling is natural green along with some evidence of yellow pigments. When cucumbers are placed in the curing brine and fermentation commences, a visible change in the color takes place.

Efforts to prevent the loss of green color in the curing process have been unsuccesful. Blanching, such as is used with other vegetable products, was tried but

although the initial green color was intensified the cucumbers lost their green color very quickly in brine.

Under the influence of acid, chlorophyll loses magnesium and forms pheophytin. In the presence of the chlorophyllase of the raw stock other chlorophyll derivatives may be formed. Willstätter and Stoll *(3)*, in their work on chlorophyll extracts from plant material, state that a metal such as copper or zinc can be introduced into pheophytin very easily by the action of their acetates upon an alcoholic solution of pheophytin to form a product similar to chlorophyll. It is generally known that copper in the presence of acetic acid will give a green pickle.

Preliminary studies were conducted to determine the effects of sodium benzoate, essential spice oils, and metallic zinc on the color of freshened cucumber stock. These showed that between 3 and 5% sodium benzoate was required in a finishing procedure to initiate a green color. This is greatly in excess of the legal limit. Likewise excessive concentrations of essential spice oils in conjunction with severe heat treatments were required to give varying degrees of green. The possibility of the oils containing sufficient copper to promote this greening was investigated. Copper contents in 25 experimental pickle samples analyzed ranged between 1 and 12 p.p.m. Of these samples, 16 showing slight green coloration averaged 3.2 p.p.m. whereas 9 green samples averaged 4.2 p.p.m. This suggested that the copper was not a factor in promoting the green color in the pickles studied.

Studies with metallic zinc-acetic acid solutions and their effect on freshened cucumber stock have shown that greening is achieved readily although some heat is required for the reaction. Concentrations of zinc and amount of heating necessary to produce greening are presently being investigated.

Acknowledgments

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Use of Antioxidants in the Frozen Storage of Turkeys[a]

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A marked reduction in rancidification of the skin and cut meat surface of turkeys in frozen storage was demonstrated by use of an aqueous gelatin coating containing antioxidants. Such a coating may be desirable whenever optimum packaging with plastic film is not economically feasible, or where large surfaces are exposed as in cut-up turkey.

The potential benefits of additions of antioxidants to fatty foods in frozen storage are well known. There remains to answer only these questions: Is rancidification in the particular commodity for its maximum storage period serious enough to make additional protection by antioxidants desirable and profitable? If so, what are the best antioxidants for the job, and what are the optimum methods of application?

In the field of poultry products, turkeys are known to be more susceptible to rancidification than chickens and, due to seasonal production, are generally stored for longer periods. Processing and storage of preponderant numbers of turkeys in eviscerated form, with exposed visceral cavity and cut surfaces, has increased the tendency toward rancidification. The now recognized (but not universally followed) optimum practices in processing and packaging should permit frozen storage life for whole eviscerated turkeys of at least one year. However, when the best packaging or processing is not economically feasible or when the carcass is cut up, the storage life is considerably reduced. In such cases, there is an obvious need for an inexpensive protective coating such as an antioxidant spray or dip might provide.

Experiments to be reported here consist of the application of several antioxidants in various carriers or solvents to the exposed surfaces of turkey steaks and half turkeys. The extent of protection in frozen storage derived from these treatments was measured by both chemical and organoleptic methods. Of necessity only a limited number of antioxidants have been tested so far, under a limited number of conditions, and the results should be considered indicative rather than decisive. Nevertheless, the data clearly demonstrate the protective effect of antioxidant dips on the skin and other exposed surfaces of turkeys.

[a] Presented at the Twelfth Annual Meeting of the IFT, Grand Rapids, Mich., June 9, 1952.

[b] Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

EXPERIMENTAL PROCEDURES

Turkeys to be used in these studies were shocked, slaughtered with an outside throat cut, slack scalded, warm eviscerated, and held in polyethylene bags at —30° F. (—34.5° C.) prior to experimental use. The frozen eviscerated carcasses were cut with a power meat saw into ⅝-inch transverse slices, and each slice was cut on a dorsal-ventral line to yield two equivalent steaks. Steaks were divided equally among treatment groups with respect to light and dark meat and with respect to location in the carcass. Half turkeys were prepared by sawing through the keel and back bone of the frozen eviscerated carcass.

In experiments with sprayed coatings, about 2 ml. of the solution of antioxidants was sprayed on the frozen steak suspended on a stainless-steel hook. An atomizer-type spray gun, made of pyrex glass and actuated by compressed nitrogen, was used. For dip coatings, the frozen steaks or half turkeys were immersed for about 2 seconds in a 3% aqueous gelatin suspension of the antioxidants, maintained at a temperature of 88° F. (31° C.). The resulting gelatin coating was about 0.2 mm. thick. All coatings were allowed to freeze before the steaks were packaged in heat-sealed M.S.A.T. cellophane bags. Steaks in individual cellophane bags were packed in locker cartons and stored at 10° F. (—12° C.) in still air. Half turkeys were wrapped in butcher paper only and stored at 10° F. (—12° C.).

Chemical analyses of the stored steaks were carried out on at least 4 steaks for each treatment. Skin and underlying fat were stripped from the steak and blended in a mixture of chloroform and anhydrous sodium sulfate to disintegrate the tissue and extract the fat. The meat was diced and dried in the frozen state and then extracted in the same manner as described for the skin fat. Chloroform extracts were analyzed for peroxides by a slight modification of the method of Wheeler *(1)*. In the case of half turkeys, peroxides were determined in the skin and underlying fatty tissue of the back.

For organoleptic evaluation, steaks were removed from frozen storage, thawed at room temperature for 1 hour, wiped free of any coating, judged for color, dipped in unseasoned flour, and grilled on a preheated plate 5 minutes uncovered on one side and 10 minutes covered on the other side. Samples of meat and fat were judged for rancid off-flavor and other off-flavor, a score of 0 signifying no off-flavor and 10 maximum or extreme off-flavor. At least 4 replications of each treatment were presented to the taste panel, which usually consisted of 6 trained persons. In some cases, due to a limited amount of skin fat, the panel was reduced to 4 persons for the evaluation of this portion of the steak. The number of treatments judged at any one taste period was limited to 4; where an experiment had more than 4 treatments, a balanced incomplete block design was used, in which each treatment was tasted with every other treatment the same number of times. Scores for other off-flavors are not reported because in no experiment were there significant differences between treatment groups. Half turkeys were evaluated by cutting the turkey halves into quarters; roasting in an electric oven at 350°F. (176.5° C.) to an internal temperature of 180° F. (82° C.) for light meat and 185° F. (85° C.) for dark meat; and scoring for rancid off-flavor in light meat, dark meat, and skin, and for rancid off-odor in light meat and dark meat.

RESULTS AND DISCUSSION

Previously reported experiments *(1)* on the frozen storage stability of antioxidant-treated eviscerated half turkeys had shown that turkeys sprayed with 0.5% solution of nordihydroguaiaretic acid in propylene glycol were more stable than turkeys sprayed with propylene glycol alone, but less stable than turkeys receiving no spray treatment. This apparent pro-oxidant effect of the sample of propylene glycol used was studied further with cross-cut turkey steaks. Although this effect is of only incidental interest in relation to the positive findings of the present report, the results will be described because of their general interest in connection with carriers for antioxidants and because of their significance in directing our attention to other carriers.

Steaks were treated as described in Table 1, packaged in heat-sealed cellophane bags, and evaluated initially and after 4 months at 10° F. (—12° C.). Initial organoleptic evaluation revealed no detectable off-flavor due to coatings. Relative differences after storage between treatment groups as measured by peroxides were essentially equivalent to differences evident from panel scores for rancid off-flavor. Deterioration was consistently greater in the propylene-glycol-sprayed steaks than in the unsprayed steaks. However, all of the steaks sprayed with propylene glycol solutions of antioxidants were superior to steaks sprayed with propylene glycol alone. Propyl gallate and butylated hydroxy anisole were roughly equivalent in their protective effects and in most cases provided steaks superior to the unsprayed controls. The effect of nordihydroguaiaretic acid was not as great, the resulting steaks being about the same as, or worse than, the unsprayed control.

Accelerated storage tests at 158° F. (70° C.) with mixtures of rendered turkey fat and various samples of propylene glycol, including the one used in the previous experiment, revealed that impurities in some samples not readily removed by redistillation were pro-oxidant in nature. However, several samples of the best commercial grade of propylene glycol, and propylene glycol prepared by hydrolysis of propylene oxide, showed no pro-oxidant effect.

A second turkey steak storage experiment was carried out, identical to the one with propylene glycol and antioxidants described above, except that refined coconut oil was used as the carrier for the antioxidants, and the concentration of antioxidant was restricted to 0.05% and that of the citric acid to 0.02% due to the limited solubility of these compounds in coconut oil. Under these conditions, no off-flavors were found initially, and no significant effects, beneficial or otherwise, were produced by the coatings and antioxidants at the relatively low concentrations used.

Since the more promising antioxidants appeared to have limited solubility in most organoleptically acceptable solvents, the possibility of using a suspension of antioxidant in a carrier was investigated. Frozen turkey steaks were dipped in 3% aqueous gelatin solutions containing in fine suspension various

TABLE 1

Effect of propylene glycol solutions of antioxidants on turkey steaks stored at 10° F. (—12° C.) for 4 months

Coating	Antioxidants in coating [c]	Peroxides, meq. per kg. fat				Rancid off-flavor scores [d]			
		Skin fat		Meat fat		Skin		Meat	
		Light	Dark	Light	Dark	Light	Dark	Light	Dark
None	None	27	17	32	31	2.0	1.6	1.0	2.1
Propylene glycol	None	86	62	45	50	2.9	3.8	1.6	2.2
Propylene glycol	0.5% NDGA and 0.5% CA	61	41	16	34	1.7	1.8	1.0	1.7
Propylene glycol	0.5% PG and 0.5% CA	25	23	12	22	1.2	1.7	0.6	0.9
Propylene glycol	0.5% BHA and 0.5% CA	14	11	12	13	1.3	2.2	0.8	0.8
Least significant difference: 5% level						1.8	1.6	0.6	1.0
1% level						2.5	2.2	0.8	1.3

[c] NDGA (nordihydroguaiaretic acid), PG (propyl gallate, BHA (butylated hydroxyanisole), and CA (citric acid) were each dissolved in propylene glycol spraying solution at 0.5% concentration where indicated.

[d] Mean values from 6 judges, 6 replications per treatment, with the 5 treatments judged three at one time, according to a balanced incomplete block design. 0, no off-flavor; and 10, maximum or extreme off-flavor.

TABLE 2

Protective effect of gelatin suspensions of antioxidants on turkey steaks stored at 10° F. (—12° C.) for 6 months

Coating	Antioxidants in coating[a]	Peroxides, meq. per kg. fat[c]				Rancid off-flavor scores[d]		
		Skin fat		Meat fat		Skin[e]	Meat[f]	
		Light	Dark	Light	Dark		Light	Dark
None	None	41	56	17	22	2.0	1.8	2.5
3% aqueous gelatin	0.2% BHA, 0.06% PG, and 0.04% CA.	16	8	8	6	1.4	0.8	1.1
3% aqueous gelatin	1.0% ascorbic acid and 4% sodium nicotinate	16	12	6	5	.....	.....	.....
3% aqueous gelatin	0.2% BHA, 0.06% PG, 0.04% CA, 1.0% ascorbic acid, and 4.0% sodium nicotinate	12	7	4	6	1.3	0.9	1.1
Least significant difference: 5% level						0.6	0.5	1.0
1% level						0.8	0.7	1.3

[a] BHA (butylated hydroxy anisole), PG (propyl gallate) and CA (citric acid) were introduced into the aqueous gelatin, at the percentages indicated, as a uniform suspension by adding a concentrated propylene glycol solution of these substances with stirring.

[c] Mean values for 8 light meat steaks, 4 dark meat steaks per treatment group.

[d] 0, no off-flavor; and 10, maximum or extreme off-flavor.

[e] Mean values, representing scores of 4 judges on skin of 16 steaks (8 light meat and 8 dark meat) per treatment group.

[f] Mean values, representing scores of 6 judges on meat of 8 light meat steaks or 8 dark meat steaks per treatment group.

combinations of antioxidants as listed in Table 2. After allowing the 0.2-0.4 mm. thick gelatin film to freeze, the steaks were packaged individually in heat-sealed cellophane bags and stored for 6 months at 10° F. (—12° C.). Mean results are summarized in Table 2.

Development of peroxides was markedly reduced by the gelatin-antioxidant coatings in all cases. In most cases the off-flavor score in the antioxidant-treated group was about one-half that found in the uncoated control. Rancid off-flavor was significantly (5% level or less of probability) reduced by the gelatin-antioxidant treatment both in the meat and in the skin. The combination of ascorbic acid and sodium nicotinate was tested because of its reported *(2)* preservative effect on the fresh color of cut meat surfaces. However, under the conditions of the test, there were no appreciable changes in surface color for any group and consequently no significant differences between treatment groups.

In order to verify the results of the latter experiment, and to determine the relative contribution of the gelatin coating without antioxidants, an additional frozen storage study on gelatin-coated turkey steaks was made. The conditions and results of this experiment are outlined in Table 3. It is apparent from either peroxide development or rancid off-flavor development that the aqueous gelatin coating alone provided some protection against rancidification. The lower concentration of antioxidants was apparently suboptimum, since it permitted greater peroxide development than the higher level. Without information for any intermediate level, the higher level of antioxidants could be used without fear of introducing off-flavors, because former tests have shown no adverse effects on initial flavor of the antioxidants at these levels. Gelatin-coated light meat steaks were scored slightly but significantly (1% level) better in appearance (absence of off-color) than the uncoated control. However, differences in off-color between coated and uncoated steaks for dark meat steaks were not consistent.

Application of the aqueous gelatin-antioxidant coating was then extended to half turkeys. The coated halves were stored at 10° F. (—12° C.) in a wrap of butcher paper in order to accelerate deterioration, and were examined after 6 months (Table 4). Although the general level of fat deterioration was greater for the halves than for the steaks, due to the packaging difference, the relative beneficial effects of the coatings for halves and steaks were in good agreement.

In interpreting results with turkey steaks in terms of eviscerated whole, half, or cut-up turkeys, it is reasonable to assume that the steak skin fat data are indicative of results that might be obtained with the skin surface of an eviscerated whole or half turkey, while the steak meat data might more closely resemble results obtained with the exposed surfaces of cut up or disjointed turkey. Since the skin fat data and the meat data lead to essen-

TABLE 3

Protective effect of gelatin suspensions of antioxidants on turkey steaks stored at 10° F. (—12° C.) for 6 months

Coating	Antioxidants in coating[j]	Peroxides, meq. per kg. fat[k]		Rancid off-flavor scores[l]		
		Skin	Meat	Skin	Meat	
					Light	Dark
None	None	71	70	2.5	2.2	2.4
3% aqueous gelatin	None	50	30	2.0	1.0	2.2
3% aqueous gelatin	0.01% BHA, 0.003% PG, and 0.002% CA	24	22	1.6	0.8	0.7
3% aqueous gelatin	0.1% BHA, 0.03% PG, and 0.02% CA	14	14	1.2	0.9	0.6
Least significant difference: 5% level				0.7	0.7	0.8
1% level				0.9	1.0	1.0

[j] BHA (butylated hydroxyanisole), PG (propyl gallate) and CA (citric acid) were added as a concentrated propylene glycol solution to aqueous gelatin with stirring to insure a uniform suspension.

[k] Each value represents average of 4 steaks.

[l] Each value for skin represents 16 steaks (8 light and 8 dark); meat values are for 8 steaks. Scoring was made on basis of 0, no off-flavor; and 10, maximum or extreme off-flavor.

TABLE 4

Protective effect of gelatin suspensions of antioxidants on half turkeys [storage in butcher paper wrap, for 6 months at 10° F. (—12° C.)]

Coating	Antioxidants in coating [m]	Peroxides,[n] meq. per kg. fat, in skin fat on back	Rancid off-flavor scores [o]			Rancid off-odor scores [p]	
			Light meat	Dark meat	Skin	Light meat quarter	Dark meat quarter
Fresh, frozen control [q]	.....	3	0.6	0.9	0.9	0.2	0.1
None	None	180	3.2	2.9	7.5	3.7	3.1
3% aqueous gelatin	None	84	2.4	2.2	7.4	3.1	3.3
3% aqueous gelatin	0.02% BHA, 0.006% PG, and 0.004% CA	48	1.5	1.4	4.4	1.9	2.2
Least significant difference at 1% level:			1.4	1.4	1.7	1.4	1.7

[m] BHA (butylated hydroxy anisole), PG (propyl gallate), and CA (citric acid) were added as a concentrated propylene glycol solution to 3% aqueous gelatin with stirring to insure a uniform suspension.
[n] Average for at least 4 turkeys.
[o] Mean values for 7 judges, 4 turkeys per treatment; 0, no off-flavor; 10, extreme off-flavor.
[p] Mean values for 4 judges, 4 turkeys per treatment; 0, no off-odor; 10, extreme off-odor.
[q] Turkeys, eviscerated and held in Cry-o-vac bags at —30° F. (—34.5° C.) prior to use.

tially the same conclusions about the protection afforded by coatings containing antioxidants, the results may have wider application than just to turkey steaks, which are, of course, a specialized and relatively undeveloped marketing form of turkey.

There is increasing evidence, from both incidental observations and controlled storage experiments, that development of objectionable rancidity of whole eviscerated turkeys in commercial frozen storage can be prevented for at least one year by careful processing and packaging in a sealed form-fitting bag made of relatively moisture- and gas-impermeable plastic. However, where optimum packaging is not feasible, or where the product has large exposed surface relative to volume, the use of some type of coating containing antioxidants merits consideration.

SUMMARY

Measurements were made of the extent of protection provided by antioxidants applied as solutions or suspensions in various carriers to the natural and cut surfaces of eviscerated turkeys in frozen storage. Peroxide development in the fat was well correlated with subjective scores for rancid off-flavor in the cooked turkey. Butylated hydroxy anisole, propyl gallate, and combinations of these antioxidants markedly reduced fat deterioration in frozen storage. Of the solvents or carriers used, a 3% aqueous gelatin solution, in which the antioxidants were suspended at about 0.15% total concentration, was found to be most effective. The 3% gelatin coating alone provided some protection, but not nearly so much as the gelatin coating containing suspended antioxidants. Pro-oxidant effects produced by some samples of solvents suggest that care should be exercised in the selection of carriers for the antioxidants. The results indicate that coatings containing antioxidants offer considerable promise for increasing the frozen storage life of turkeys, especially when large surfaces are exposed as in eviscerated and cut-up poultry.

Acknowledgment

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The Effect of Fluctuating Storage Temperature on the Quality of Stored Frozen Foods[a,b]

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The effects of food temperatures fluctuating between 0° F. (—17.7° C.) and 10-13° F. (—12.2° to —10.5° C.) on the quality of frozen ground beef, ground pork, strawberries, and snap beans stored for periods up to 10 months were measured. In addition, comparisons were made between these effects and those caused by storage time, wrapping material, shape of package, and storage at a constant temperature of 0° F.

The studies reported in this paper were part of a larger project initiated by the Bureau of Human Nutrition and Home Economics of the U. S. Department of

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Agriculture to determine the effect of fluctuating temperature on the quality of stored frozen foods, as a basis for establishing performance requirements for home freezers. The project was carried on cooperatively by the Bureau and the Agricultural Experiment Stations of Minnesota and Iowa, and Cornell University.

Work at Minnesota was begun on October 1, 1946. The purpose of these studies was to determine whether fluctuating storage temperature, within the range used in the experiments, may be a major factor affecting changes in the quality of frozen ground beef, ground pork, strawberries, and snap beans.

To accomplish this purpose it was necessary to evaluate the significance of other variables normally encountered in the storage of frozen foods. In order to accentuate any effects of fluctuating temperatures, the cycles were arranged so that the resulting temperature changes in the product might be much greater than normally would be encountered under commercial, locker-plant, or home-freezer storage conditions.

The literature includes relatively few data about the effects of fluctuating temperatures on stored frozen foods. Hustrulid and Winter *(8)* reported in 1943 no deterioration of palatability in snap beans, corn, peas, strawberries, raspberries, and cantaloupe, stored for 6 months at food temperatures fluctuating from 0° to —20° F. (—17.7° to —28.8° C.) as compared with a constant storage temperature of about 5° F. (—20.5° C.).

Gortner, Fenton, Volz, and Gleim *(7)* stored pork roasts, strawberries, snap beans, and peas for 12 months at 0° F. (—17.7° C.) at 10° F. (—12.2° C.) and at fluctuating 0° to 20° F. (—17.7° to —6.6° C.). They found that the quality changes (palatability and ascorbic acid) of foods stored at 10° F. were similar to those occurring at the fluctuating temperatures. The quality of these foods stored at 0° F. was definitely superior to that of the foods stored at 10° F. and at fluctuating 0° to 20° F. They suggested that exposure of the foods to temperatures above 0° F. rather than merely fluctuating temperatures may have been the major factor in these quality changes.

Hustrulid, Winter, and Noble *(9)* found that temperature fluctuations below 0° F. (—17.7° C.) were not important in the deterioration of color, texture, and flavor of snap beans, strawberries, ground beef, and ground pork or in ascorbic acid loss in strawberries. However, desiccation of meat wrapped in waxed locker paper was much greater under fluctuating storage conditions than at constant 0° F. There was no significant difference in desiccation when the meat was wrapped in laminated aluminum foil.

Pottinger *(10)* found that palatability scores and volatile acid numbers (for showing relative freshness) for mackerel fillets stored at fluctuating temperatures of 0° to —10° F. (—17.7° to —23.3° C.) fell between these for fillets held at constant 0° F. and constant —10° F. Similar results were reported in comparing storage at fluctuating 0° to 15° F. (—17.7° to —9.4° C.) temperatures with constant 0° F. and constant 15° F.

Emerson, Brady, and Tucker *(5)* found that storage of beef for periods up to 6 months at 0° F. (—17.7° C.), 10° F. (—12.2° C.), and fluctuating temperatures between these ranges did not materially affect the palatability, moisture losses or rate of autolysis when a good moisture-vapor proof packaging material was used. On the other hand, Woodroof and Shelor *(12)* reported that strawberries, blackberries, raspberries, and peaches deteriorated more rapidly when stored at temperatures fluctuating from 0° to 10° F. (—17.7° to —12.2° C.) than at a constant temperature of 10° F.

EXPERIMENTAL METHODS

The storage temperatures, storage periods, and products studied during 1946, 1947, and 1949 are shown in Table 1. The effects of temperature, storage time, wrapping material, and shape of package were measured and the significance of these variables was determined by statistical analysis of variance.

Storage units. An 18-cubic foot, 2-compartment, chest-type home freezer manufactured by the Seeger Refrigerator Company of St. Paul, Minnesota, was used for keeping the samples at a "constant" temperature of 0° F. (—17.7° C.). The adjustment of the controls was such that the unit operated on a short cycle. For this reason the air temperature in any part of the storage space remained fairly constant.

A home-built, chest-type refrigerator with a single-compartment storage volume of about 35 cubic feet was used for the fluctuating-temperature storage of the 1946-47 experiments.

In 1947-48 and 1949-50, an 18-cubic foot, 2-compartment, chest-type freezer manufactured by the Victor Products Company of Hagerstown, Maryland, was used for fluctuating 0° to 10° F. (—17.7° to —12.2° C.) storage. In order to obtain the necessary temperature fluctuation, thermostatic temperature control was added to this freezer. By means of a double-pole, double-throw switch, it was possible to use either the added

TABLE 1

Storage temperatures, storage periods, and products packed, 1946-50

Year packed	Storage temperature	Storage period	Products packed
		months	
1946	Constant 0° F. (—17.7° C.)	3 to 4,[c] and 6	Strawberries, snap beans, ground pork, ground beef
	Fluctuating 0° F. (—17.7° C.) to 10° F. (—12.2° C.)	3 to 4,[c] and 6	Strawberries, snap beans, ground pork, ground beef
1947	Constant 0° F. (—17.7° C.)	4, 8, and 10	Strawberries, ground beef, ground pork
	Constant —40° F. (—40° C.)[e]	4, 8, and 10	Strawberries, ground beef, ground pork
	Fluctuating 0° F. (—17.7° C.) to 10° F. (—12.2° C.)	4, 8, and 10	Strawberries, ground beef, ground pork
1949	Constant 0° F. (—17.7° C.)	4, 4,[d] and 10	Ground beef, ground pork
	Constant —25° F. (—31.6° C.)[e]	4, 4,[d] and 10	Ground beef, ground pork
	Fluctuating 0° F. (—17.7° C.) to 10° F. (—12.2° C.)[e]	4, 4,[d] and 10	Ground beef, ground pork
	Fluctuating 0° F. (—17.7° C.) to 13° F. (—10.5° C.)[f]	4, 4,[d] and 10	Ground beef, ground pork

[c] Beans and pork judged at 3 month period, strawberries at 4 months. Beef not judged at this period.

[d] Held at constant temperature of —25° F. for 6 months, then at fluctuating temperatures of 4 months, but data not usable due to breakdown of equipment in the —25° F. storage.

[e] Data not usable because of accidental power failure and breakdown of equipment during the storage period.

[f] Planned for 0° to 15° F. but actual temperatures averaged 0° to 13° F.

control or the one furnished with the original equipment. The added thermostatic control governed the operation of the unit on the "high" temperature setting and the original equipment governed it on the "low" temperature setting.

A Coldspot home freezer was used for fluctuating 0° to 13° F. (—17.7° to —10.5° C.) storage during 1949-50.

Temperature fluctuations. During 1946-47, the food temperature was fluctuated through 3 cycles per week as shown by Figure 1 except for some holidays. The method was to set

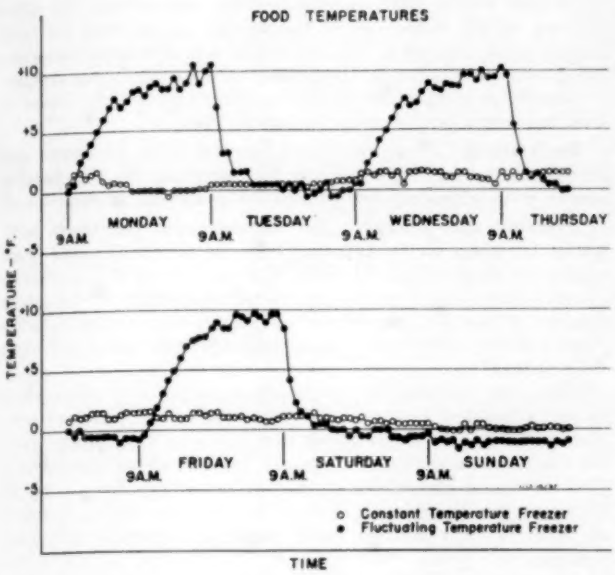

Figure 1. Typical weekly record of food temperatures at constant and fluctuating temperatures, 1946-47.

the control "up" one morning and to set it "down" the next. A rough calculation of average food temperature for the week of January 13-19 gave 3.5° F. (—15.8° C.). The arithmetic average of the fluctuating temperature for the whole period would be somewhere between 3° and 4° F. (—16.1° to —15.5° C.).

Figure 1 shows also that there were no very rapid or large changes in temperature of the food stored in the "constant temperature" freezer. The temperature did vary in the range 2° F. (—16.6° C.) to —2° F. (—18.8° C.) over a rather extended period. The control setting was never changed but the storage temperature was influenced slightly by variations in room temperature.

In order to simulate more nearly the actual temperature situation in a home freezer being used in a home, a different method of fluctuating the temperature was used during 1947-48. For the most part the food was subjected to the following sequence of fluctuations per week: 3, 2, 1, 0, or a total of 6 fluctuations per month. Also, the length of time for which the food was subjected to the higher temperature was reduced. As shown by Figure 2 (top), the method of operation was to set the temperature control "up" in the morning (8 A. M.) and down again in the evening (5 P. M.). The total time from normal storage temperature 0° F. (—17.7° C.) up to 10° F. (—12.2° C.) then back to 0° F. was planned to be between 15 to 20 hours. The shape and character of the temperature curve will depend upon the load in the freezer and the setting (differential) of controls. The effective load was kept at about 100 lbs. of ice by adding ice as food was removed for judging.

Two freezers were used for fluctuating storage temperatures during 1949-50, one for 0° to 10° F. (—17.7° to —12.2° C.) and the other planned for 0° to 15° F. (—17.7° to —9.4° C.). The same sequence of temperature fluctuations was used during 1947-48 and 1949-50.

Figure 2 (lower) shows one cycle of temperature fluctuation for the 0° to 15° F. (—17.7° to —9.4° C.) freezer. In this the food obviously did not reach 15° F. A single control was used on this freezer making it difficult to regulate each cycle to reach 15° F. The high temperatures were in the ranges 11° to 17° F. (—11.6° to —8.3° C.) with an average of about 13° F. (—10.5° C.).

The temperatures were recorded with a Leeds and Northrup resistance-type, 16 point, —30° to 50° C., recorder. The accuracy is about ±0.5° C. The nickel temperature-sensitive elements were frozen into water in containers of the same size and shape as used for strawberries and beans.

Supplemental food temperature measurements were made with a portable potentiometer using iron-constantan thermocouples and with liquid-in-glass thermometers.

Packaging and wrapping materials. Waxed frozen food containers with disc lids, 8-ounce capacity, were used for snap beans and similar containers of 16-ounce capacity for strawberries. In 1946 and 1947 the ground meat was wrapped in a standard 48-pound waxed locker paper and also in a laminated paper comprising 0.00035 gauge aluminum foil laminated to kraft paper. In 1949, only the waxed locker paper was used.

Ground meat. Special effort was made to obtain ground meat of good quality and uniform character. Beef packed in 1947 and 1949 for panel scoring was ground from forequarters, grade U. S. Good, aged at about 38° F. (3.3° C.) for 7 and 9 days, respectively. There was no record of the aging period in 1946. Pork was ground from shoulder butts of hogs butchered less than 48 hours previously. The pH of the pork used in 1946 was 6.0 after grinding.

Each lot of meat was ground twice through a power grinder and in each instance the grinder was carefully washed before use. The ground meat was given an additional mixing by hand and then placed in a cold-wall-type mechanical refrigerator during the time required for processing (usually not more than 2 hours). No salt or seasoning of any kind was added. In 1946 and 1947 the beef was formed by means of a wooden mold, approximately 6 inches long, 4½ inches wide and ¾ inch in depth, into solid rectangular blocks which averaged about 300 g. in weight. The pork was formed into circular patties, each approximately 2 1/16 inches in diameter and 7/16 inch in depth and weighing about 28 g. Then 6 patties were packed in one layer in each package.

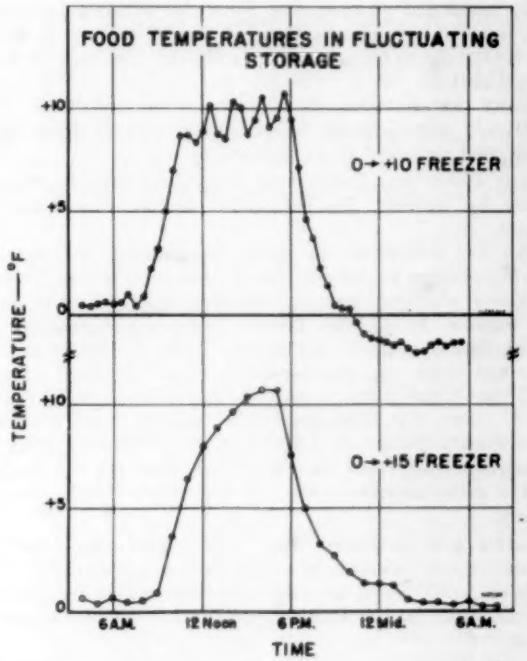

Figure 2. Typical daily record of food temperatures at fluctuating temperature, 1947-48 and 1949-50.

In 1949, the meat was wrapped in two distinct shapes, designated as "stacked" and "flat" as shown in Figure 3. The latter method was the same as used for pork in 1946 and 1947. The "stacked" method consisted of wrapping 3 patties in a single package, one on top of the other but separated by a sheet of waxed paper. The patties were formed in a special circular mold, each 4 inches in diameter and ½ inch thick. The exposed surface area of the "flat" method in relation to volume was approximately three times that of the "stacked" method.

In 1946 and 1947, a double butcher-type wrap was used for all the waxed paper wraps and a single wrap of the same type for the laminated foil wraps. In 1949, a double butcher-type wrap was used for the "stacked" method and a double druggist-type wrap for the "flat" method as it was difficult to apply the same method of wrapping to both types of package. Sheets measuring 12 by 14 inches were used for all wraps. All folds were secured by pressure-sensitive locker tape.

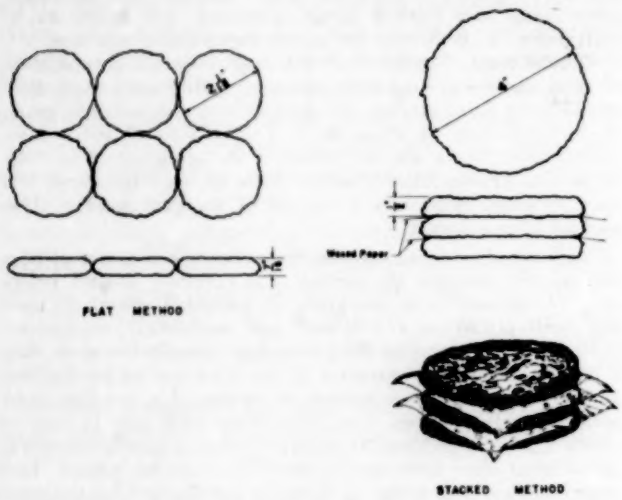

Figure 3. Size and distribution of meat patties in "flat" and "stacked" methods before wrapping, 1949-50.

Snap beans and strawberries. Kentucky Wonder beans were broken into 1¼ inch lengths, blanched for 3½ minutes in boiling water, cooled for an equal period in ice-water, and then packaged in waxed cartons.

Premier and Catskill strawberries (1946 and 1947 packs, respectively) were washed, hulled, mixed with ⅓ their weight of sugar, and packaged in waxed cartons.

Color. Color was determined objectively by the Munsell whirling disc method. The desirability of color was judged by the panel.

Under the conditions of these experiments, the value of measuring changes in color of the ground meat is open to question. Color changes, due to oxidative reactions, may occur rather rapidly during the various processing operations and after the thawed meat is unwrapped. Allen *(1)* shows that the oxygen and water vapor permeability of the wrapping material exert a significant influence on the color of fresh beef. Robertson *(11)* states that fresh meat color and meat color stability will sometimes change at different rates in different wrapping materials and that color may be readily lost (in the wrapped package) if the package is not sent directly to the freezer after wrapping.

Ascorbic acid determinations. The frozen snap beans and strawberries were blended in a Waring blendor with 3% metaphosphoric acid. Ascorbic acid was determined on the centrifuged extract by the Bessey photoelectric indophenol method *(3)*.

Press fluid and moisture of meat. Press fluid was determined by the method of Child and Baldelli *(4)* for 6 replicate samples. Moisture was determined by the difference in weight of triplicate samples before and after heating for 18 hours in an oven at 60° C. under 25 inches of mercury.

Weight records. The net weight of each meat sample packed in 1946 was determined by recording the weight of wrapping material used for each sample and deducting this from the gross weight of each package after wrapping was completed. Weighings at the end of the storage period were made at low ambient temperatures to avoid errors due to condensation of moisture on the cold surfaces. A Cenco agate-bearing, trip-type balance with a sensitivity of about 0.1 g. was used.

Weight losses were determined by subtracting the total package weight at the end of the storage period from the total weight at the beginning. No correction was attempted for possible uptake of moisture by the packaging material during storage.

Similar procedures were used to determine weight losses of snap beans and strawberries packed in 1946.

Replications. Four replicated lots for each treatment and period of storage were packed for sampling by the judging panels, with additional lots packed for analytical studies.

Freezing and storage. All packages and containers were placed promptly on the freezing plates of an upright-type home freezer at about —5° F. (—20.5° C.) as soon as the processing of each series of 15 to 20 units was completed. Within about 2 hours or less the products were transferred to a nearby locker plant freezing room which was equipped with two heavy-duty 24-inch fans.

After not less than 24 hours, the products were transferred to the scheduled storage conditions where the packages were distributed loosely in the lower part of the storage space where the temperature gradient was small. Special effort was made in 1949 to obtain uniformity of storage conditions by placing all packages one layer in depth on a wire screen, with a space of at least ¼ inch between each package, vertically and horizontally.

Preparation for judging. The ground meat, after thawing, was broiled in the broiler compartment of a kitchen range. In the 1949 series, 4 patties were selected at random from each "flat" package and formed into a single patty of the same size as one in the "stacked" package. Thus all the meat actually judged was in patties of about the same size and weight (about 114 g.). The middle patty in a "stacked" package was separated from the outside patties and the palatability judged as a function of position in the package. Only one of the two "outside" patties, chosen at random, was used from each package.

The frozen snap beans were placed in a small quantity of boiling water, covered, and cooked until tender.

The strawberries were thawed in the package in front of an electric fan until the ice crystals had melted.

Judging. Judges were selected on the basis of their experience and knowledge of foods and on their availability for judging at the scheduled hours. Panels of 6 persons were used in 1946 and 1947, and of 5 persons in 1949. The same panel judged all lots of the same product packed at the same time. Three of the judges served on all panels during 1946-49.

The meat packed in 1946 and 1947 was judged for color, visible desiccation, tenderness, juiciness and flavor. In addition, aroma was scored for the 1947 pack. Color and flavor only were judged for the 1949 pack. The snap beans were judged for flavor, color, texture, and shape. All these factors were scored on a basis of 0 to 10, with 10 being the highest possible score.

RESULTS

Meat

Ground beef and pork. Mean palatability scores for flavor, aroma, and desiccation of beef and pork packed in 1946 and 1947, together with a summary of the variables statistically significant at the 1% level, are given in Table 2. Similar data for flavor and color of beef and flavor of pork packed in 1949 are given in Table 3.

TABLE 2

Mean palatability scores [a] for ground beef and pork stored at constant 0° F. (—17.7° C.) and fluctuating 0° to 10° F. (—17.7° to —12.2° C.) temperatures, 1946 and 1947 packs

Palatability factor	Product	Year packed	Storage period	Waxed paper wraps		Laminated foil wraps		Variables significant at 1% level
				Constant temperature	Fluctuating temperature	Constant temperature	Fluctuating temperature	
			months	*scores*	*scores*	*scores*	*scores*	
Flavor	beef	1946	6	6.0	3.7	6.1	6.4	temperature [b, i] wrapping material
		1947	4	8.0	6.8	8.8	8.8	storage period
		1947	8	5.9	3.8	6.9	6.1	wrapping material
		1947	10	5.4	3.2	5.4	5.9	temperature [b]
	pork	1946	3	8.8	8.3	8.9	8.5	storage period
		1946	6	7.7	5.1	7.9	7.1	temperature [i] wrapping material
		1947	4	7.7	7.4	8.8	8.5	storage period
		1947	8	5.8	3.3	7.1	5.9	temperature
		1947	10	6.2	3.6	6.5	5.3	wrapping material
Aroma	beef	1947	4	8.3	8.2	8.8	8.9	storage period
		1947	8	6.9	5.9	7.0	6.7	wrapping material
		1947	10	6.0	4.2	6.3	6.7	
	pork	1947	4	8.4	8.5	9.0	8.6	storage period
		1947	8	6.6	5.7	6.8	7.0	temperature
		1947	10	6.9	5.9	6.6	6.3	
Desiccation (visible)	beef	1946	6	7.5	3.6	9.4	9.1	temperature wrapping material
		1947	4	7.5	5.9	9.6	9.3	storage period [b]
		1947	8	3.8	3.5	9.1	9.2	wrapping material
		1947	10	3.9	3.7	9.0	8.1	
	pork	1946	3	8.0	7.0	9.5	9.7	temperature [i]
		1946	6	7.9	5.1	9.2	9.1	wrapping material
		1947	4	7.1	7.5	9.6	9.6	storage period
		1947	8	5.4	4.9	9.0	8.9	wrapping material
		1947	10	6.0	4.0	8.8	8.5	

[a] Four replicate samples scored by 6 judges. Mean scores before freezing were 9.0 or higher for flavor and aroma, 10.0 for desiccation.
[b] Significant at the 1% level for the waxed paper wrap only.
[i] Data from a parallel project with fluctuating 0 to —10° F. (—17.7° to —23.3° C.) temperatures are included in the statistical analysis.

Scores for color of pork and tenderness and juiciness of beef and pork are not included in these tables because of disagreement among judges as indicated by a statistically high significance of the variances between judges. Data on color of beef is given only for the 1949 pack to indicate the differences due to shape of package and position in the package.

Flavor of meat. Beef and pork packed in 1946, 1947, and 1949 were scored for desirability of flavor. Both waxed locker paper and laminated foil were used in 1946 and 1947, but only waxed locker paper was used in 1949. Two different shapes of packages were used in 1949, as previously described.

The data for the 1946 and 1947 packs, as summarized in Table 2, show that storage period and wrapping material were consistently significant at the 1% level. Differences between constant and fluctuating temperatures were not as consistently significant as were differences due to storage period and wrapping material, except in the waxed locker paper wraps. In 1946, the

TABLE 3

Mean scores [j] for flavor of ground beef and pork, color of ground pork stored at constant 0° F. (—17.7° C.) and fluctuating 0° to 13° F. (—17.7° to —10.5° C.) temperatures, 1949 pack

Factor	Product	Storage period	Inside patty in "stack"		Outside patties in "stack"		"Flat" shape of package		Variables significant at 1% level
			Constant temperature	Fluctuating temperature	Constant temperature	Fluctuating temperature	Constant temperature	Fluctuating temperature	
		months	*scores*	*scores*	*scores*	*scores*	*scores*	*scores*	
Flavor	beef	4	7.8	7.7	6.7	8.1	6.5	7.7	shape of package
		10	5.7	6.0	6.1	6.6	4.7	3.7	storage period
	pork	4	8.5	8.5	8.7	8.3	8.2	7.7	position in "stack"
		10	6.7	7.4	5.2	6.0	5.3	4.6	shape of package storage period judges
Color	beef	4	6.3	5.1	8.1	7.4	7.9	7.7	position in "stack"
		10	6.0	4.7	6.8	4.6	4.0	3.3	shape of package storage period storage temperature

[j] Four replicate samples scored by 5 judges. Mean scores before freezing were 9.5 to 9.7 for flavor; initial color of beef was good but no scores were recorded.

statistical analysis included a parallel series of experiments with temperature fluctuations of 0° to —10° F. (—17.7° to —23.3° C.).

The flavor values remained relatively constant during the first 3 to 4 months of storage. With the 1947 pack, when 3 storage periods were used, there was an appreciable decline in desirability of flavor between the 4- and 8-month period. Thereafter, there was little additional decrease. At the 8- and 10-month periods, flavor values for the constant 0° F. (—17.7° C.) temperature were much superior to the fluctuating 0° to 10° F. (—17.7° to —12.2° C.) level except for beef wrapped in laminated aluminum foil.

With the 1949 pack, where two shapes of packages were used, the differences between the "flat" and "stacked" packages were more consistently significant than those due to any other variable except time of storage. The "stacked" package was much superior, as shown in Table 3, after a storage period of 10 months.

In the "stacked" packages of pork the position of the patty was significant at the 1% level, the inside (middle) patty of the three being much superior in flavor to the outside patties. Banks *(2)* reports comparable results in a study with herring frozen in blocks where the peroxide value of herring at the surface of a block was always higher than that of the remainder of the herring in the same block. There was no significant difference with beef in the position of the patty.

The data, as summarized in Tables 2 and 3, show that the constant 0° F. (—17.7° C.) temperature was consistently superior to the fluctuating temperature except with beef wrapped in laminated foil and with beef and pork in the "stacked" packages.

Aroma of meat. Beef and pork packed in 1947 were scored by the judging panel for aroma. The scoring was done immediately after the meat was removed from the broiler and placed on the table for judging. The data, as summarized in Table 2, show that differences between constant and fluctuating temperatures were not as consistently significant as were differences due to storage period, except in the waxed locker paper wraps. There was little if any significant difference due to storage temperature when a laminated foil wrap was used.

Desiccation of meat. Beef and pork packed in 1946 and 1947 were scored by the judging panel for visible desiccation. The data show that the wrapping material was by far the most important variable under all conditions. The constant 0° F. (—17.7° C.) storage temperature was superior to the fluctuating temperature in most instances when the meat was wrapped in waxed paper, but there was little if any difference when a laminated foil wrap was used.

These findings are supported by the data on weight losses recorded with the 1946 pack of beef and pork as shown in Table 4. Weight losses under the fluctuating storage temperature for the waxed paper wraps were more than three times as great as those where the meat was held at a constant temperature. Losses for the meat wrapped in laminated foil were negligible and were independent of storage conditions.

Moisture and color of meat. No significant change in moisture in the 1946 pack of beef and pork was found due to storage time, temperature, or wrapping materials. Desirability of color in the frozen product after thawing appeared to be affected by two separate and distinct factors, (a) darkening of the surface color of the product, presumably as a result of oxidative changes, and (b) the appearance of a dull gray surface color resulting from desiccation.

TABLE 4

Weight losses of frozen ground beef and pork stored for 6 months at constant 0° F. (—17.7° C.) and fluctuating 0° to 10° F. (—17.7° to —12.2° C.) temperatures, 1946 pack

Product	Weight loss in waxed paper wrap[k]		Weight loss in laminated foil wrap[k]	
	Constant temperature	Fluctuating temperature	Constant temperature	Fluctuating temperature
	%	%	%	%
Beef	1.58	5.46	0.33 (gain)[l]	0.08
Pork	1.94	6.14	0.11	0.37 (gain)[l]

[k] Average of 6 samples.
[l] Apparent slight gain may have been due to absorption of moisture by the wrapping material.

The apparent influence of oxidative changes was clearly indicated when "stacked" packages were used in 1949 for beef wrapped in waxed locker paper, a relatively permeable wrap. The color of the most exposed surfaces (outside patties in the "stack" and all patties in "flat" packages) was markedly superior to the surface color of the middle patty in the "stack" at the 4-month storage period. Dull gray surface colors were responsible for the low scores in most of the lots after storage for 10 months, especially those stored at fluctuating temperatures.

Tenderness of meat. Beef and pork packed in 1946 and 1947 were scored for tenderness. The variances between judges were statistically highly significant at all times, this factor apparently being the most important of all variables with the 1946 pack of pork and second in importance with the 1946 pack of beef and the 1947 pack of pork. The data thus indicate the difficulty of obtaining reliable and consistent results in evaluating tenderness by means of a judging panel owing to differences between judges as to what constitutes tenderness. For this reason, tenderness was not scored in judging the 1949 packs.

Juiciness of meat. Beef and pork packed in 1946 and 1947 were scored for juiciness. As with tenderness, the variances between judges were statistically highly significant at all times, this factor apparently being the most important of all variables with the 1946 pack of pork and second in importance with the 1946 pack of beef. The problem of obtaining exact uniformity in doneness in broiling the meat is a difficult one to overcome. Juiciness was not scored in judging the 1949 packs because of the indicated difficulties involved in obtaining reliable data.

Press fluid of meat. No significant change in press fluid in the 1946 pack of beef and pork was found due

TABLE 5

Mean judging scores [a] and ascorbic acid content of snap beans and strawberries stored at constant 0° F. (—17.7° C.) and fluctuating 0° to 10° F. (—17.7° to —12.2° C.) temperature

Factor [b]	Storage period	1946 pack				Storage period	1947 pack	
		Snap beans		Strawberries			Strawberries	
		Storage temperature		Storage temperature			Storage temperature	
		Constant	Fluctuating	Constant	Fluctuating		Constant	Fluctuating
	months					*months*		
Flavor (by judging panel)	0	8.3	8.3	8.2	8.2	0	8.1	8.1
	3 or 4 [c]	8.7	8.6	8.0	7.1	4	8.1	8.2
	6	8.1	7.9	8.2	7.6	8	6.4	5.4
						10	6.2	5.0
Color (by judging panel)	0	8.2	8.2	8.2	8.2	0	8.5	8.5
	3 or 4	8.7	8.3	7.6	7.7	4	8.1	7.7
	6	8.0	8.0	8.3	8.2	8	7.6	6.4
						10	6.2	6.2
Texture (by judging panel)	0	8.2	8.2	6.8	6.8	0	6.9	6.9
	3 or 4	8.4	8.5	6.5	6.8	4	7.6	7.2
	6	8.0	7.8	7.7	7.2	8	5.9	5.6
						10	5.6	5.0
Shape (by judging panel)	0	8.7	8.7	7.4	7.4	0	8.0	8.0
	3 or 4	8.9	8.8	7.2	7.2	4	8.6	7.7
	6	8.5	8.6	8.0	7.7	8	7.2	6.8
						10	5.9	5.9
Ascorbic acid (by indophenol method) mg. per 100 g.	0	9.0	9.0	34.8	34.8	0	52.5	52.5
	3 or 4	8.1	6.9	32.0	27.1	4	43.6	44.7
	6	6.5	5.1	32.1	25.6	8	43.6	47.0
						10	46.3	44.1

[a] Four replicate samples were scored by 6 judges.
[b] Maximum score for each factor was 10.
[c] The beans were stored for 3 months, the strawberries for 4 months.

to storage time, temperature, or wrapping material. Gaddis, Hankins, and Hiner *(6)* have recently reported no significant relationship between the percentage of press fluid and scores for juiciness with beef, lamb, mutton, and chevon.

Effect of shape of package. The flavor of the beef and pork in the "stacked" packages, which had the least surface area of meat in relation to volume, was much superior to that in the "flat" packages after storage for 10 months.

Strawberries and Snap Beans

As shown in Table 1, strawberries and green beans were used in 1946-47 and strawberries in 1947-48 to study the effect of storage at constant 0° F. (—17.7° C.) and at temperatures fluctuating between 0° and 10° F. (—17.7° to —12.2° C.).

The beans and the 1946 pack of strawberries were given similar scores by the judges regardless of the storage conditions. Thus, as may be seen from Table 4, the average scores for the flavor, color, texture, and shape of the various samples usually fell between 7.5 and 8.5 out of a possible 10 points, regardless of whether the samples had been stored at the fluctuating or at the constant temperature, or whether they were judged just after freezing or 6 months later. Neither the temperature nor the storage means were significantly different, according to an analysis of variance.

The judges' results for color were corroborated by Munsell color disc readings, which showed no consistent changes in hue, value, or chroma due to differences in temperature conditions or storage periods.

The ascorbic acid content was slightly higher when the samples were stored at the constant than at the fluctuating temperature, and at 3-4 months storage than at 6 months. Although the differences between the means were small, they were statistically significant.

The 1947 pack of strawberries received higher scores when stored at 0° F. (—17.7° C.) than at 0° to 10° F. (—17.7° to —12.2° C.). The mean values for flavor and color were significantly different at the 1% level, those for texture and shape at the 5% level. The scores decreased, moreover, with increase in storage period, the differences for all quality factors being significant at the one per cent level.

These findings are not necessarily contradictory to those for the 1946 crop, although at first thought they may appear to be. The reason is that, as may be seen from Table 5, the first significant lowering of scores came at the 8-month storage period. By then the berries had been stored 2 months longer than any of the 1946 crop.

The mean ascorbic acid content of the 1947 berries was not significantly different at the constant and at the fluctuating storage temperatures. A loss of ascorbic acid occurred during the first 4 months of storage, but not after that. These results are contradictory to those obtained for the 1946 brries, and the reason for the difference is not clear. The 1947 samples showed much more variation in ascorbic acid than the 1946 ones. Such variability would mask as slight a trend as the 1946 samples showed.

Weight losses at the fluctuating temperature, as shown in Table 6, were more than 4 times as great with beans and more than 3 times as great with strawberries as those where the products were held at a constant temperature.

TABLE 6

Weight losses of frozen snap beans and strawberries stored for 6 months at constant 0° F. (—17.7° C.) and fluctuating 0° to 10° F. (—17.7° to —12.2° C.) temperature

Product[P]	Year packed	Loss of weight when storage temperature was	
		Constant	Fluctuating
		%	%
Snap beans	1946	0.26	1.12
Strawberries	1946	0.34	1.26

[P] Average of 18 samples for snap beans, 12 samples for strawberries.

SUMMARY

Ground beef, ground pork, strawberries, and snap beans were stored at a constant temperature of 0° F. (—17.7° C.) and at fluctuating temperatures between 0° to 10° F. (—17.7° to 12.2° C.) and 0° to 13° F. (—17.7° to —10.5° C.) for periods up to 10 months. During 1946-47 the temperature was fluctuated through 3 cycles per week, and during 1947-48 and 1949-50 through 6 cycles per month.

In general, differences between the constant and fluctuating temperatures in flavor, aroma, and desiccation of ground beef and pork were much less than the differences due to storage time, wrapping material, and shape of package.

Differences between the ground meat stored at constant and fluctuating temperatures were greatly influenced by the packaging material and by the shape of the package. Only one type of container was used in the studies with strawberries and snap beans.

When wrapped in waxed locker paper, ground beef and pork stored for 6, 8, or 10 months at the constant temperature were scored significantly higher in flavor, aroma, and in absence of visible desiccation, than when stored for similar periods at the fluctuating temperatures. In most instances, a similar trend was apparent at the 3- and 4-month storage periods. There was little, if any, difference between the constant and fluctuating temperatures at all storage periods, except for desirability of flavor of pork, when a wrap of laminated aluminum foil was used.

Loss of weight was much greater in the ground meat stored at fluctuating temperature when wrapped in waxed locker paper, but when wrapped in laminated foil the losses were negligible at both constant and fluctuating temperatures.

The flavor of beef and pork patties wrapped in compact form in waxed locker paper and stored for 10 months at constant and fluctuating temperatures was significantly more desirable than that of patties wrapped in a single layer which had a much larger exposed surface area in relation to volume. The difference in flavor scores due to shape of package was much greater than that between the constant and fluctuating temperatures. The flavor of the pork from the "inside" patties was significantly more desirable than that from the "outside" patties.

Strawberries stored for 8 and 10 months at a constant 0° F. (—17.7° C.) temperature were scored significantly higher in flavor, color, texture, and shape than when stored for similar periods at a fluctuating 0° to 10° F. (—17.7° to —12.2° C.) temperature. However, neither strawberries nor snap beans when stored for 6 months or less showed a significant difference in quality. The ascorbic acid content was only slightly, if at all, affected by the different storage temperatures and periods. Loss of weight was much greater at the fluctuating temperature.

In instances where higher scores were given to products stored at a constant temperature, the data do not indicate whether the results might be due to a more even temperature or to a lower average value at the constant temperature.

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A Comparison of Various Methods for the Determination of Spoilage in Fish[a,b]

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A number of chemical methods have been evaluated as indicators of various stages of spoilage of such west coast fish as tuna, mackerel, and sardines (pilchards). The determinations of total volatile and trimethylamine nitrogen, hydrogen sulfide, indole, oil acidity, and carbonyl compounds, as bisulfite-binding substances, were of very limited significance. The value of the content of steam volatile acids and of volatile reducing substances was investigated in more detail. Of all the methods tried, that of the volatile reducing substances gave the closest correlation with the condition of the raw and canned fish as judged organoleptically.

Spoilage in meat and fish has been investigated for many years. Of the many possible types of deleterious changes included in the term "spoilage," those brought about by microorganisms are of particular interest to handlers of proteinaceous products. Bacterial action on meat and fish gives rise to volatile breakdown products and these in turn are responsible for marked odors that develop during spoilage. This has led to the widespread use of the sense of smell and of other related organoleptic criteria to determine the state of preservation of fish and other flesh foods. However, organoleptic tests are subjective. Their use is limited by the individual's sense acuity, physiological condition, mood, and preference, as well as by environmental and atmospheric conditions at the time of sampling.

REVIEW OF METHODS

To eliminate the inherent limitations in the organoleptic procedures and yet retain the basic principle, many attempts have been made to use chemical and other tests to measure the changes associated with bacterial spoilage and the development of odoriferous products. Among the tests proposed as spoilage indicators are those for total volatile nitrogen, mono-, di-, and trimethylamine *(1, 2, 3, 16, 19, 23)* indole *(8, 9, 23)*, hydrogen sulfide *(4)*, volatile acids *(6)*, oil acidity *(5)*, steam volatile oxidizable substances *(12, 13, 14, 17)* and oxidizable substances in protein-free filtrates of aqueous extracts of meat, fish, and fat *(22)*. A bioassay method has been reported *(11)*. Reviews of the spoilage literature may be found in papers by Reay and Shewan *(18)*, Sigurdsson *(20)*, and Soudan *(21)*. None of the tests have proved of sufficient general usefulness to warrant their widespread adoption. The probable explanation is that a large number of microorganisms with different biochemical activities are involved in spoilage. The type of bacterial spoilage in a particular food will thus depend upon the kinds of bacteria in the environment at a particular time. Furthermore, different types of foodstuffs and even various kinds of a single foodstuff may give rise to different classes of odoriferous products with the same bacterial contamination. For example, bottom fish, such as cod or haddock, may develop an ammoniacal odor whereas tuna may give rise to a sour or otherwise unpleasant odor.

The data in Table 1 illustrate the variation in biochemical activities of bacteria isolated in this laboratory from fish from various sources. Relatively few organisms formed indole. At most only a little more than half of them split fats and liquefied gelatin, and about two-thirds of the most active produced hydrogen sulfide. Any chemical method dependent solely upon the presence of any one of these products could be expected to yield negative results in many instances, even though bacterial action had taken place.

TABLE 1

Variation in biochemical properties of organisms isolated from various sources

Source	Organisms producing			
	Lipolysis	H_2S	Liquefied gelatin	Indole
	%	%	%	%
Sardine spoilage experiment	53	20	53	27
Sardine spoilage experiment	37	20	30	30
Bluefin tuna spoilage experiment	39	39	32	0
Bluefin tuna spoilage experiment	38	20	30	30
Tuna from ice boats	23	67	16	19
Tuna from brine boats	45	45	21	0

Work on the detection and prevention of spoilage in fish was started at the Fisheries Research Laboratory in the summer of 1937. The immediate problem was tuna. The fishermen had extended their cruising radius for tuna without making any provision to preserve the fish on the boats; losses of raw tuna rose as high as 25 to 50% and on occasion even to 100%. In addition, tuna was often rejected after being precooked and sometimes after being canned. The financial loss from all tuna rejected due to spoilage in 1937 was approximately $750,000. The report that follows mainly presents the results of spoilage runs carried out since then in an effort to find an objective precise method of determining the extent of spoilage in commercial fish products.

EXPERIMENTAL RESULTS

At the start, chemical and other methods proposed as useful indexes of spoilage were surveyed and many of them tested. Table 2 shows the data for total volatile

[a] Based on a paper presented at the Marine Products Symposium, Division of Agricultural and Food Chemistry, at the 114th Meeting of the A.C.S., September 13, 1948.

[b] Work carried out as part of the activities of the Fisheries Research Laboratory, consultant to the California Department of Public Health and supported by the Fish Packing Industry of California.

TABLE 2
Volatile nitrogen compounds in raw tuna loins during storage at various temperatures

Hours stored	Nitrogen per 100 ml. press juice as							
	Total volatile, when stored at			Trimethylamine, when stored at				
	68° F.	70° F.	75° F.	68° F.	70° F.	72° F.	75° F.	82° F.
	mg.	*mg.*	*mg.*	*mg.*	*mg.*	*mg.*	*mg.*	*mg.*
0	....	....	38.8	....	....	0.2	0.6	1.0
2.8	....	43.3	....	....	1.7	....	....	....
3.7	30.5	....	....	1.2	....	....	....	0.7
5.2	....	....	35.7	....	....	0.4	0.9	....
9	....	40.5	34.2	....	1.6	0.6	1.4	1.4
13	....	....	32.1	....	....	0.6	1.0	0.8[c]
17	....	37.2	33.0[c]	....	2.2	0.8	1.9[c]	0.5
18.3	36.6	....	....	1.3	....	....	....	....
21.5	....	39.1	32.8	....	1.5	2.0	4.4	0.5
26.8	36.3	....	....	2.3	....	....	....	0.8
29	39.6[c]	41.0[c]	....	3.2[c]	3.4[c]	....	....	....
46	....	....	....	....	....	....	....	2.6

[c] Values indicate where samples organoleptically were judged spoiled.

TABLE 3
Content of substances associated with spoilage in bluefin tuna after storage in brines at various temperatures and in ice

Storage temperature		Brine concentration	Indole		H_2S[e]		Ammonia[f]		Trimethylamine[f]		Carbonyl compounds[e]	
°C.	°F.	%	Loin	Belly	Loin	Belly	Loin	Belly	Loin	Belly	Loin	Belly
			γ per 100 g.	*γ per 100 g.*								
—1.1	30	3.5[d]	0.3	3.0	0	0	9.5	10.3	0	0	55.5	248.6
—5.6	22	10.5	1.5	0.3	0	0	7.5	5.2	0	0	44.1	107.6
—20.6	—5	23	0	0.2	0	0	5.2	8.6	0	0	16.5	21.2
—1.1	30	Ice	0.3	0.8	2.7	..	10.7	..	0.43	..	56.0	..

[d] Fish at 30° F. (—1.1° C.), in ice were spoiled after 30 days, in brine after 47 days. Others were still passable after 50 and 57 days.
[e] Ml. 0.01 *N.* iodine solution per 100 g.
[f] Mg. nitrogen per 100 ml. of press juice.

and trimethylamine nitrogen obtained during separate spoilage experiments on raw tuna. The values corresponding to samples judged spoiled by organoleptic means are underlined in this and in subsequent tables. Similar results were found for California sardines and Pacific mackerel. The data for a storage experiment on raw tuna in brines and in ice at various temperatures are given in Table 3, in which indole, hydrogen sulfide, volatile nitrogen compounds, and carbonyl compounds were determined. The latter substances were determined by an adaptation of the bisulfite-binding substances method of Clift and Cook *(7)*.

The values for total steam volatile acids produced during a number of tuna spoilage experiments are summarized in Table 4. A spoilage run on raw tuna during storage in various brines and in ice at various temperatures was carried out. The data for the content of steam volatile acids and of carbonyl compounds in the canned samples corresponding to the various raw ones are shown in Table 5. The steam volatile acids in commercially canned tuna and Pacific mackerel of different degrees of freshness are exemplified by the data of Table 6. In Table 7 are given the values found for steam volatile acids in raw Pacific mackerel during spoilage at various temperatures.

TABLE 4
Steam volatile acids in raw tuna loins during storage at various temperatures

Storage time at 54° F.	0.01 *N.* acid per 100 g.	0.01 *N.* acid per 100 g. when stored at				
		Storage time	68° F.	70° F.	75° F.	82° F.
days	*ml.*	*hours*	*ml.*	*ml.*	*ml.*	*ml.*
0	20.0	0	....	....	20.0	....
4	17.0	2.7	....	15.9	....	....
5	25.0	3.8	17.1	....	....	....
....	....	5.5	....	....	14.2	9.8
6	32.5	8.0	....	....	....	13.0
7	41.0	9.3	....	17.5	....	14.8
8	73.0[g]	12.0	....	....	....	20.8[g]
9	74.5	14.0	....	....	18.4	....
10	81.0	16.5	....	20.0	....	28.3
14	105.0	18.0	19.3	....	20.6[g]	....
		21.0	....	17.7	30.3	28.0
		27.0	16.6	....	....	....
		29.0	26.2[g]	21.3[g]	....	....
		46.0	....	....	....	....

[g] Values indicate where samples organoleptically were judged spoiled.

TABLE 5
Volatile acids and carbonyl compounds in tuna canned after raw storage

Storage temperature		Storage medium	Odor of can	Volatile acids 0.01 *N.* per 100 g.	Carbonyl compounds 0.01 *N.* iodine solution per 100 g.
°C.	°F.			*ml.*	*ml.*
—1.1	30	3.5% brine[h]	Bad	30.6	60.0
		10.0% brine	Fair, slightly strong	20.1	19.8
—5.6	22				
—12.2	10	17.0% brine	Good	17.0	15.1
—20.6	—5	23.0% brine	Good	13.5	12.1
—1.1	30	Ice	Slightly strong, not good	20.0	14.2
—5.6	22	Ice	Slightly strong	15.1	16.5
—12.2	10	Ice	Fair	16.9	9.7
—20.6	—5	Ice	Fair	19.4	10.5

[h] Fish in 3.5% sodium chloride at —1.1° C. (30° F.) were stored 48 days, others 56 days.

On the basis of the above and of similar results from other experiments, it was concluded that the substances tested as spoilage indicators were of limited or no significance for the species of fish encountered in our work. All of the chemical methods proposed and tried out depend upon the production and presence of either a single compound or of a specific group of compounds

TABLE 6

Steam volatile acids (SVA) in commercially canned tuna and Pacific mackerel

Odor [1]	Species of Tuna	SVA 0.01 *N.* per 100 g.	
		Tuna	Mackerel
		ml.	*ml.*
Good	Bluefin	19.3	14.7
		20.9	17.6
		.....	18.8
		.....	17.1
	Yellowfin	14.3	19.5
		17.8	.....
"Sweet"		.....	19.8
		.....	19.0
		.....	19.6
		.....	18.3
Slightly strong but passable	Bluefin	20.7	.....
		19.0	.....
Strong		.....	16.4
		.....	18.0
		.....	15.0
		.....	18.9
Stale	Yellowfin	23.1	19.9
		15.6	17.0
		.....	16.6
		.....	17.2
Slight taint		.....	18.4
Taint	Yellowfin	19.9	.....
		20.8	.....

[1] Organoleptic grades for tuna determined by professional fish smeller.

TABLE 7

Steam volatile acids in raw Pacific mackerel during storage at various temperatures in the round and eviscerated

Storage time	0.01 *N.* acid per 100 g. when stored			
	Round at			Eviscerated at 68° F.
	68° F.	76° F.	78.5° F.	
hours	*ml.*	*ml.*	*ml.*	*ml.*
1.5	8.7	.....	6.8	8.3
6.0	.....	.....	19.5	.....
7.2	.....	9.7	16.8	.....
9.0	.....	.....	16.8	.....
10.0	.....	14.3[1]	.....	.....
11.0	12.0	.....	19.5[1]	10.8
12.8	.....	19.5	26.0	.....
16.0	17.8	.....	.....	12.5
25.5	17[1]	.....	.....	15.3[1]

[1] Values indicate where samples organoleptically were judged spoiled.

As the range of biochemical activities of the bacteria found in fish is wide, as illustrated in Table 1, at one time a specific compound may be present and at another time it may be absent during spoilage.

A chemical method as reliable as the organoleptic criteria of spoilage was sought. A procedure was developed *(15)* which does not depend upon the presence of any specific compound or type of compound but reflects the presence of a wide variety of volatile oxidizable or reducing substances.

Laboratory air is cleaned by passage through distilled water, 3 bottles containing approximately 1.5 *N.* potassium permanganate in 2.5 *N.* sodium hydroxide solution, followed by 2 spray traps. With 2 cubic feet (about 72 liters) of the washed air, measured by a flowmeter, the volatile substances from a sample of fish press juice are aspirated in 40 minutes into the oxidizing reagent, ordinarily an 0.02 *N.* solution of potassium permanganate in 1 *N.* sodium hydroxide solution. The amount of potassium permanganate reduced by the volatile substances, expressed as microequivalents, is used as the measure of the content of volatile reducing substances (VRS) in the sample. Two reaction vessels each containing 10 ml. of the oxidizing agent are used per sample.

The sample is prepared by pressing a suitable amount of raw fish or the entire contents of a can to obtain the press juice; this is centrifuged to yield a clear aqueous portion and 5 ml. of the latter are used as the sample. A control for unreacted reagent is set up at the same time as the test (10 ml. of each) and the difference between the control and the test titrations represents the amount of oxidizing reagent reduced. The residual potassium permanganate is determined by acidifying the reagent with 5 ml. of 6 *N.* sulfuric acid, adding 3 ml. (an excess) of 20% potassium iodide in 0.1% sodium carbonate solution and titrating the liberated iodine with about 0.025 *N.* sodium thiosulfate solution, using starch solution as the inside indicator. A single unit and a portable 2-unit VRS apparatus may be seen in *(10)*; a number of these units may be set up, all connected through a manifold and a vacuum storage tank to a vacuum pump.

To test the efficacy of the volatile reducing substances method as an index of the state of preservation of fish, a large number of storage experiments were carried out on different kinds of fish at various temperatures. Determinations were made on both raw and canned samples after increasing storage times. Table 8 shows the increase in volatile reducing substances in raw tuna loins during storage at various temperatures. The experimental material is in part similar to that in Table 4. In Table 9 are seen data for canned tuna samples corresponding to some of the raw samples in Table 8. A summary of the spoilage of raw Pacific mackerel is given in Table 10. In Table 11 are shown the VRS values in Pacific mackerel canned from the raw material of Table 10. Representative data for VRS values

TABLE 8

Volatile reducing substances in raw tuna loins during storage at various temperatures

Storage time at 54° F.	Microequivalents reduction per 5 ml. press juice	Storage time	Microequivalents reduction per 5 ml. press juice when stored at			
			64°F.	68° F.	70°F.	75°F.
days		*hours*				
0	11.5	0.0	.....	.....	.....	10.8
5	13.7	2.5	8.9	.....	.....	.....
6	16.2	3.7	.....	10.4	.....	.....
7	23.0[k]	5.5	13.4	.....	.....	12.8
8	37.5	9.3	.....	.....	17.0	14.5
9	38.8	13.5	.....	.....	.....	18.7
14	41.8	16.0	15.4	.....	24.6	.....
		18.0	.....	.....	.....	24.3[k]
		21.5	.....	17.8	23.4[k]	32.8
		28.5	20.1	25.5[k]	29.5	.....
		39.5	22.4[k]	.....	.....	.....
		51.5	27.8	.....	.....	.....
		61.5	59.2	.....	.....	.....

[k] Values indicate where samples organoleptically were judged spoiled.

TABLE 9

Volatile reducing substances in canned tuna packed from fish stored in the round at various temperatures

Storage time	Microequivalents reduction per 5 ml. press juice when stored at			
	64° F.	68° F.	70° F.	75° F.
hours				
0	.....	.....	.....	8.4
2.5	.....	.....	11.5	.....
4.0	12.5	6.7	.....	.....
5.1	.....	.....	.....	13.4
7.5	11.7	.....	.....	.....
9.5	.....	.....	12.8	15.0
13.5	.....	.....	.....	18.2
17.0	9.0	.....	12.5	20.1[l]
18.5	.....	10.5	.....	.....
21.5	.....	.....	.....	24.2
25.0	.....	13.3	.....	.....
29.0	17.6	13.6	.....	.....
39.5	25.0[l]	.....	.....	.....
61.5	27.3	.....	.....	.....

[l] Values indicate where samples organoleptically were judged spoiled.

together with some for pH and oil acidity, in commercially canned Atlantic mackerel, kippered shad, and kippered salmon are shown in Table 12. Data for VRS values in raw California sardines during storage in the round at various temperatures are shown in Table 13.

An individual spoilage experiment showing various determinations on California sardines canned in brine during storage in the round at 68°F. (20° C.) is shown in Table 14. In Table 15 are brought together VRS values for California sardines canned in brine after progressive storage in the raw state. Similar data for California sardines canned in tomato sauce are shown in Table 16. The results of a spoilage experiment on squid during storage in still and running seawater are given in Table 17.

TABLE 10

Volatile reducing substances in raw Pacific mackerel during storage at various temperatures in the round and eviscerated

Storage time	Microequivalents reduction per 5 ml. press juice when stored			
	Round at			Eviscerated at 68° F.
	68° F.	76° F.	78.5° F.	
hours				
3	17.2	15.8	6.2	18.0
5	.....	17.5	.....	.....
6.8	.....	.....	18.5	.....
7.5	.....	19.2	25.5[m]	.....
10	17.3	45.5[m]	.....	.....
11.3	.....	.....	22.5	.....
15	.....	.....	67.5	.....
16	19.5	.....	.....	18.8
25.5	30.8[m]	.....	.....	24.8[m]

[m] Values indicate where samples organoleptically were judged spoiled.

TABLE 11

Volatile reducing substances in canned Pacific mackerel packed from raw fish stored under varying conditions

Storage time	Microequivalents reduction per 5 ml. press juice when stored			
	Round at			Eviscerated at 68° F.
	68° F.	76° F.	78.5° F.	
hours				
1.5	14.0	13.5	13.9	14.0
4.5	.....	14.6	.....	.....
6.0	.....	.....	14.7	.....
7.0	.....	16.1	17.1	.....
9.0	.....	18.0	17.1	.....
11.0	12.6	.....	19.8[n]	13.9
12.0	.....	20.6[n]	.....	.....
15.0	14.7	.....	34.4	15.8
25.0	26.1[n]	.....	.....	16.0
31.0	.....	.....	162.4	.....

[n] Values indicate where samples organoleptically were judged spoiled.

TABLE 12

Volatile reducing substances (VRS) in commercially canned Atlantic mackerel, kippered shad, and kippered salmon

Organoleptic judgment	pH for mackerel	Oil acidity ml. 0.1 *N.* per ml. oil for salmon	VRS Microequivalents reduction per 5 ml. press juice		
			Mackerel	Shad	Salmon
Normal odor and appearance	6.06	2.03	12.7	22.2	34.8
		2.20	.....	37.8	40.6
		.....	.....	28.2	33.4
		.....	.....	39.9	.....
		.....	.....	22.2	.....
		.....	.....	18.6	.....
		.....	.....	19.2	.....
		.....	.....	25.2	.....
Stale, soft, honeycomb in mackerel	6.10	.....	30.9	45.0	55.3
Taint, meat discolored, honeycomb	6.21	2.59	36.5	71.4	65.6
	5.85	2.50	58.7	95.4	74.2
		3.87	.....	102.3	75.8
		2.26	.....	111.3	74.6
		.....	.....	.....	114.6
		.....	.....	.....	193.0
		.....	.....	.....	212.7

TABLE 13

Volatile reducing substances in raw California sardines (pilchards) during storage in the round under various conditions

Storage time	Microequivalents reduction per 5 ml. press juice when stored at					
	45° F.[p]	56° F.	Dry 56° F.	64° F.	Dry 68° F.	Dry 70° F.
hours						
0	.....	21.0	23.0	8.0	25.0	12.0
10.0	4.5	27.5	.....	29.0	.....	21.0
13.0	.....	.....	.....	.....	36.0	.....
16.5	.....	.....	25.0	.....	.....	.....
21.5	.....	.....	38.0[o]	.....	.....	.....
23.0	11.0	7.5	.....	24.5	40.0[o]	46.0[o]
24.5	.....	.....	39.0	.....	.....	.....
27.0	.....	16.0	.....	.....	.....	91.0
29.5	.....	.....	41.0	.....	.....	.....
30.5	.....	.....	44.5	.....	.....	.....
32.0	.....	43.0[o]	.....	.....	.....	.....
33.5	.....	60.0	.....	42.5[o]	106.0	.....
40.0	35.5[o]	.....	.....	.....	.....	.....
41.0	.....	.....	.....	62.5	.....	.....
45.0	.....	.....	100.0	.....	.....	.....
57.5	.....	103.0	.....	.....	.....	.....
71.0	83.0	.....	.....	.....	.....	.....

[o] Values indicate where samples organoleptically were judged spoiled.
[p] All fish were stored in seawater except those marked "dry."

TABLE 14

Increase in volatile reducing substances, steam volatile acids, and oil acidity in canned sardines in brine

Raw fish stored in seawater at 20° C. (68° F.)	Condition of can contents	VRS Microequivalents per 5 ml.	Steam volatile acids ml. 0.01 *N.* per 5 ml.	Oil acidity mg. KOH per ml.
hours				
0	Acceptable	6.9	3.92	0.775
6.25	Acceptable	5.5	3.76	0.754
10.25	Acceptable	7.6	3.33	0.809
18.75	Acceptable	15.1	4.70	0.921
25.5	Not acceptable	33.0	7.20	1.024
29.75	Not acceptable	76.4	5.96	1.580

TABLE 15

Volatile reducing substances in canned California sardines in brine backed from fish stored in the round under varying conditions

Storage time	Microequivalents reduction per 5 ml. press juice when stored at					
	56° F.[r]	Dry 56° F.	64° F.	Dry 66° F.	Dry 68° F.	Dry 70° F.
hours						
0	10.0	10.0	.....	.....	10.0	.....
8.5	12.0	.....	.....	.....	.....	.....
10.5	.....	.....	.....	5.5	.....	.....
11.5	6.0	.....	7.0	.....	9.0	12.0
16.5	.....	13.0	.....	.....	.....	.....
18.5	.....	.....	.....	14.0	.....	.....
20.0	.....	13.0	.....	.....	.....	44.0[q]
22.0	17.0	15.0	13.0	.....	11.5	.....
25.0	.....	21.5[q]	.....	33.0[q]	.....	.....
27.5	27.9[q]	27.0	.....	.....	.....	83.0
30.0	.....	.....	.....	72.0	.....	.....
31.0	36.0	30.5	.....	.....	.....	.....
34.0	38.5	.....	43.8[q]	.....	42.0[q]	.....
37.5	42.0	.....	.....	.....	.....	.....
40.0	.....	.....	88.0	.....	.....	.....
45.0	.....	80.0	.....	.....	.....	.....
49.0	.....	.....	.....	.....	.....	96.0
58.0	82.0	.....	104.0	.....	.....	.....

[q] Values indicate where samples organoleptically were judged spoiled.
[r] All fish were stored in seawater except those marked "dry."

TABLE 16

Volatile reducing substances in canned sardines in tomato sauce packed from fish stored in the round under varying conditions

Storage time	Microequivalents reduction per 5 ml. press juice when stored at				
	56° F.[t]	Dry 60° F.	64° F.	Dry 68° F.	Dry 70° F.
hours					
0	11.5	19.5	15.0	22.0	17.5
11.0	.....	20.0	13.0	20.0	.....
20.0	.....	20.5	.....	52.0*	63.0*
23.0	.....	.....	23.0	.....	.....
25.0	15.0	.....	.....	.....	.....
26.5	.....	.....	.....	82.0	.....
33.5	16.0	.....	46.0*	.....	.....
40.0	.....	32.5	71.0	.....	.....
42.0	30	.....	.....	.....	.....
49.0	46.5*	54.0*	.....	.....	.....

* Values indicate where samples organoleptically were judged spoiled.
[t] All fish were stored in seawater except those marked "dry."

TABLE 17

Volatile reducing substances in squid stored in still and running seawater at 58.3° and 54.1° F., respectively

Storage time	Microequivalents reduction per 5 ml. press juice when stored			
	in still water		in running water	
	Raw	Canned	Raw	Canned
hours				
0	6.5	13.2	6.5	13.2
4.0	4.7	10.3	4.8	8.1
13.5	11.0	13.1	8.1	10.0
22.5	71.7*	54.2*	63.4*	17.0
30.0	82.1	76.4	75.8	24.4*
38.0	93.3	85.4	89.3	43.4
49.0	.....	96.9	.....	61.8

* Values indicate where samples organoleptically were judged spoiled.

DISCUSSION

For evaluating early stages of spoilage the measurement of indole, volatile nitrogen compounds, hydrogen sulfide, and pH is of no practical significance for such fish as tuna, California sardines, and Pacific mackerel. The steam volatile acids content is a variable quantity, which apparently depends largely upon the type of spoilage microorganisms predominating during the deterioration. For canned fish the steam volatile acids, oil acidity, and carbonyl compounds methods were of much less significance than for raw samples.

The method depending upon the measurement of volatile reducing substances gave best correlation with the state of preservation of either raw or canned fish, as judged organoleptically. In this procedure the very reactive alkaline potassium permanganate solution acts only on the constituents of the sample which are volatile at room temperature and does not come into contact with the mixture of substances present in the sample of press juice, extract of the fish or in a steam distillate. The original material is subjected to as little manipulation and contact with extraneous reagents as possible.

The VRS content shows an increase during storage of the various fish species at different temperatures. All canned samples judged spoiled organoleptically had a higher VRS content than those judged passable. California sardines stored in running seawater at 56° F. (12° C.) at first lost some of their content of volatile reducing substances. However, even in this case the VRS content definitely increased with the onset of spoilage. The sardines stored dry in their own accumulating juices at 56° F. (12° C.) were from the bottom of a load of fish, whereas those stored at the same temperature in seawater were from the top of the load. The latter spoiled about 10 hours after the former.

The data in Table 12 show that the VRS procedure is applicable to products such as kippered shad and salmon, which received a treatment imparting an added aroma and flavor. The distinction between normal and not passable samples is evident.

Certain basic requirements must be met by a chemical method generally applied as a spoilage index: The method must be at least as sensitive as organoleptic criteria; hence the results must correlate very closely. It must be able to detect the early stages of spoilage, when organoleptic criteria often yield doubtful results and subjective factors tend to confuse the judgments. It must be as simple as possible from an operational point of view and enable rapid determinations to be made.

On the basis of the experimental data accumulated to date, it is believed that the volatile reducing substances procedure most closely fits these specifications. It does not depend upon the persence or production of any specific compound or class of compounds, but will detect any substance which is volatile in air and which will reduce or be oxidized by alkaline potassium permanganate solution. Whatever the spoilage pattern may be, as long as the deterioration of the fish is accompanied by the production of odors and hence of volatile substances, they will be detectable by the volatile reducing substances procedure. The organoleptic means of evaluating foodstuffs is at best only roughly quantitative, but even slight changes in the state of samples can often be detected with the volatile reducing substances procedure before they become very evident to the senses.

The aeration portion of the procedure has been standardized to last 40 minutes. Including the time required to prepare the sample and set up the apparatus, the volatile reducing substances content in a sample can be determined within 60 to 65 minutes. If a multiple unit apparatus is used, many more values can be obtained within practically the same time. A steam volatile acids determination, for example, is much more time-consuming and complex from the standpoint of manipulation.

A sample of canned tuna where an aromatic essential oil had been added to the vegetable oil used in canning was submitted to the laboratory. The aqueous portion of two cans had a volatile reducing substances content of 8.1 and 9.6 microequivalents of reduction per 5 ml., the oily portions had 100.5 and 78.0 microequivalents per 5 ml. The added essential oil was detectable by the volatile reducing substances method in the oily portion; the aqueous portion had a volatile reducing substances content within the normal range.

This method is being used in the examination of samples of commercially packed fish in order to evaluate the freshness of the material used in the pack. Several thousand cans of sardines, tuna, mackerel, and other fish in both experimental and commercial packs have been examined. On the basis of these data volatile re-

ducing substances values for canned fish packed from raw material of various degrees of freshness have been established. With biological material the sampling procedure may greatly influence the results obtained, as the source material is generally not homogeneous and uniform. This is also true for raw fish, and therefore the resulting canned product may be expected to vary from can to can. To ascertain the condition of a given lot, at least 6 cans and usually 12 or more are examined. Using this method of evaluation, the following ranges of volatile reducing substances values for various types of canned fish products are tentatively proposed, keeping in mind the fact that biological material may not be homogeneous.

Microequivalents reduction per 5 ml. of press juice

Species and style of pack	Acceptable	Borderline or questionable acceptability	Not acceptable
Sardines and mackerel in brine	17 and under	17 to 20	Above 20
Sardines and mackerel in tomato sauce	35 and under	35 to 40	Above 40
Tuna	15 and under	15 to 20	Above 20

These ranges have been found useful in judging many commercially canned fish samples. As more data accumulate, the ranges may need revision.

The volatile reducing substances (VRS) procedure can be looked upon as a means of chemically evaluating the amount of odoriferous substances in a sample, regardless of subjective interpretation. Some data illustrating more general application to other foods and food products have been published *(10)*.

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What a Management Consultant Can Do for a Client Organization[a]

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The two methods of classification of consultants are defined. The contributions of consultants to the solution of business problems are discussed. Examples are given of actual consulting assignments and the qualities of a good consultant are noted.

To students of economic history, one of the most important and significant phenomena in the long-term growth of the American economy is the way in which fields of endeavor spring up and expand in response to demonstrated needs. Often these concerted movements far outstrip the rest of the economy as they become recognized and accepted.

Since the shooting part of World War II ceased in 1945, there has been a number of these phenomena on the American scene. Probably the best known of these is television, which has zoomed from practically nothing to a multi-billion-dollar industry in the short space of 7 years. Another is the rebirth of investment trusts. Currently, they are filling a need to find practical investment outlets for the lower and middle income groups.

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Another and less well publicized expansion has occurred in the consulting field, particularly since 1945. Although consultants have been operating effectively for many years, the emergency of World War II gave them a chance to demonstrate their worth to managements that had not previously used consultants as need after need arose for competent professional assistance.

An evidence of greater use of management consulting services can be seen in the growth of the Association of Consulting Management Engineers, familiarly known as ACME. This organization was founded in 1929 to develop and maintain high standards of ethical practice in the profession. Although representing only a small percentage of the total number of consulting firms in the country, ACME has gained general recognition as the central body representing the profession today. Membership is widely accepted as evidence of stability, reliability, and competence.

In 1945, this organization was composed of 26 member firms. Today, it has 38, an increase of 46%. Even more significant, the firms have grown larger and matured. This is shown by an increase of more than 100% in the number of principals, that is, top executives, in these firms. This steady growth gives no signs of letting up.

This development is further indicated by looking at the amount of annual national income derived from engineering and professional services other than legal and medical. Between 1945 and 1950, the latest year reported by the Department of Commerce, national income from this source rose from 409 to 856 million dollars, a more than a 100% increase, while total national income was advancing about 31%. In other words, the rate of gain in consulting since the war has been more than 3 times faster than the growth of our economy expressed in dollars.

I wish, at this time, to dispel some of the confusion which inevitably arises in the minds of clients who are about to engage an outside consultant for the first time. It will be helpful to spell out some definitions and some classifications. A consultant is an individual, or in the plural, a group of people joined in a firm to provide professional advice, assistance, or services. The basic principle of consulting is sound. On the one hand, it enables operating organizations who have a need for skilled assistance to secure such help for a minimum period of time to do a specific job. On the other, it enables consultants to spend full time in their field and thereby secure a depth of penetration into scientific management which they could not otherwise do if they also held operating responsibilities. The potential value and economy to business of such a service is obvious.

Two ways in which consultants can be classified are into "general management consultants" and into "specialty consultants." In general, the management consultants are larger firms which work across the board in providing counseling on all of the major functions of business, including organization, sales and marketing, finance and accounting, manufacturing and engineering. These firms as a rule also provide counsel to a broad range of business activities and industries.

"Specialty" consulting firms normally have one or more specialties in which they function. Example of these would be firms which work in the area of public relations, industrial relations, marketing, finance and accounting systems, manufacturing, and the like. There are also specialty consulting firms which work in particular industries. As a general rule, specialty firms are smaller than the management consulting firms but many of them are sound professionally and have a record of excellent results.

Our firm is in management consulting. It is broad both in the functions and in industries served. Approximately one half our business is in top organization, management, and general surveys and the other half in surveys of specific operating problems and installations. Although my comments will be based primarily on management consulting experience, I believe they are essentially applicable to any type of professional consulting relationship.

WHAT CONSULTANTS CONTRIBUTE TOWARD THE SOLUTION OF A BUSINESS PROBLEM

What can consultants provide over and above what operating men can do for themselves in solving a business problem? In this regard I will discuss 5 specific points.

An unbiased and objective viewpoint. In some instances people have been in considerable difficulty because they didn't have an objective, unbiased viewpoint. Take as an example an individual acting as president and sales manager, both of which should be full-time jobs, not doing either one justice.

In working on a particular assignment, the consultant's only objective is to develop a workable solution. In doing this, he must be completely detached from the conditions and personalities in the particular situation. He does not have to contend with the problems of long-time friendships or with historical habits or fixed work patterns of particular industries or companies.

Trained analysts. A competent staff of a consulting firm must consist of men who have general problem solving ability and training. They must know how to gather and assemble facts, make a sound analysis, and develop a practical solution to a business problem. The members of such a staff are selected for native abilities of this type, receive additional training and gain practical experience on every working assignment. On the other hand, within a client's organization, it is not unusual to find a very successful operating man who does an outstanding job of administering or heading a department who does not have any special analytical ability.

Staff with specific experience. Consultants are also selected for the experience they have acquired previously. In addition to the background experience they have when they come with the firm, in their consulting experience they are faced time after time with similar problems in particular related areas. I should point out that this does not mean in any way that such men should use a canned approach in solving a problem. There is a vast difference between having a pat program which is installed time after time and in using real creative thinking based on a sound background of experience in developing or creating a solution which is particularly suited to a selected set of conditions.

Uninterrupted application of time. The normal procedure for a consulting firm in developing a solution to a business problem is to apply a particular staff on an uninterrupted basis to the problem. The advantages of this approach and the results which accrue from it, it seems to me, are the ones least often recognized. There is no question but that this approach certainly does save elapsed time in arriving at a solution. However, there is considerably more to it than this. A man who keeps at a task steadily invariably completes it with fewer man-hours expended than does a man of equal ability who can work on it only spasmodically.

Ability to present ideas and obtain action. The consultant's success is generally measured in terms of the results growing out of his association with a client. This means that the end product of the consulting relationship is constructive action on the part of the client enterprise's management, not developing a program or submitting a report as such.

It is the consultant's responsibility to develop sound solutions, and to interpret and explain them clearly and forcefully. The provocation for engaging a consultant is the existence of a management problem. At the outset of an assignment, both the client and the consultant are aiming at a course of action that meets the needs of the situation. Management's confidence in the solution emanates in part from the soundness and logic of the solution itself and also from the manner in which it is presented. If management does not have the confidence to take action, the professional assignment is not a successful one.

Presenting reports to management involves formal written reports as well as oral discussions. The oral presentation lends itself to accomplishing understanding of broad concepts. The written report supplies the detail of the analysis and supporting data. The written report is useful as a guide to installation of a program involving a number of steps that might require a period of time for their accomplishment.

Both written reports and oral discussions are strengthened through the use of visual aids. Charts, diagrams and tables are helpful in presenting ideas so that they can be readily understood.

The consultant's position as an outsider in presenting a program permits him to open up the thinking of client personnel at any level to accomplish thorough consideration. He is not restricted by protocol. He can get a hearing for ideas that cannot safely or effectively be done by members of the client organization.

These five points, then, are the special contributions which consultants can make to the solution of a business problem.

HOW ASSIGNMENTS WITH CONSULTANTS SHOULD BE ARRANGED

In order to clarify what these points mean in actual practice, it is worthwhile to run through the basic steps of a typical consulting assignment.

Defining the problem involves recognition of the problem by both the client and the consultant and a mutually developed definition of its scope and limits. We always include the definition of the problem in a specific proposal letter which also covers the following points:

1. Definition of the amount and kind of talent the client will provide for the assignment. It is important to note that most assignments require at least some time of management personnel and also time of technical or specialized personnel.
2. Specific plan for accomplishing the study including estimated amount and types of consulting talent required, and also estimated elapsed time.
3. Definition of what the consultant's responsibility in the project will be and what product the consultant will present to the client.
4. An estimate of charges, both for professional time and out-of-pocket expenses.

There are a few points worthy of note relating to the conduct of the assignment. This, of course, varies for the wide range of problems covered, but for a typical assignment consists of the following:

1. Obtain and assemble all pertinent facts relative to the area under study.
2. Analyze all facts and develop basic solution to the problem.
3. Organize results and present to management basic solution.
4. Develop details of solution to problem or recommended program.
5. Assist operating organization in installation of changes or program.
6. Prepare and submit report on an assignment.
7. As required or requested, assist in continuation or audit effectiveness of program.

HOW CONSULTING ASSIGNMENTS WORK OUT IN PRACTICE

To illustrate these principles, I will run through two examples of actual assignments which have been conducted by our firm.

Medium-size company. This company manufactured consumer hard goods such as small appliances, kitchenware, etc. This is a relatively old firm with a good record of sales and profits until recent years when the profit ratios have been dropping and other problems have been cropping up.

We had made a general survey, which is not the subject of my example. During the survey it became apparent to our manufacturing staff that, in addition to whatever other problems there were, its manufacturing methods and procedures were out of date and in bad shape. As a result, manufacturing costs were out of line and it was virtually impossible to meet competition's costs. As a result of the over-all survey, one of our major recommendations was that an industrial engineering department be installed to carry on a long-term continuous methods and cost reduction program.

The management requested our help in the engineering department installation.

We developed with the management of the company a statement of objectives for the industrial engineering department, determined where it should fit in the organization, and developed the basic organization pattern for the industrial engineering department itself. We were able to find within the company a man who had the basic capacity and most of the experience required to be chief industrial engineer. After the management had made the arrangements with this man to take on the job, we developed with him a specific training program to fill in the areas where he lacked practical experience. We also found two other men in the organization to be industrial engineers and assisted in bringing in two more from the outside.

As a trial run, two methods assignments were selected. Joint teams composed of the members of the department and the members of our staff were formed to carry these assignments through to completion.

By the time they were finished, the client had a functioning industrial engineering department properly placed in their organization with a clear understanding of its objectives. In this particular instance, the savings which accrued from the two methods projects paid for our work on this assignment.

Small division of large corporation. This division had a product which had been under development for several years. The problem was whether or not it should be launched on the market.

A member of our staff with both engineering and sales background was assigned to this project. He started out on the assignment pretty much on the basis of making the usual marketing survey. However, our man soon found out that the product had certain basic and inherent shortcomings. As a result of this finding, we spent only half of the estimated time on the job but we did give the client a comprehensive answer to the problem. From the data we had gathered we were able to give him a rough estimate of the potential market for a real practical product such as the one on which he was working. Secondly, we told him that unless he was in a position to spend and gamble specific amounts of money to whip the design problem and to carry on an adequate merchandising program, he should drop all work on this particular product.

WHAT TO LOOK FOR IN A CONSULTANT

In order to accomplish results such as those illustrated by the examples, the management consultant

should bring to the client specific attributes. High ethical standards are basic, as is confidence in the confidential, professional handling of an engagement. The consultant should provide a creative approach, his analysis and his programs should be tailored to the specific situation of the individual client operation. Practical answers are essential; the programs suggested must be constructive and workable. Good management of his own affairs on the part of the consultant is important to do the job he agrees to do at a high quality standard within the estimates of time and cost. A consultant must be able to establish smooth working relationships with client people at all levels. He must have a constructive, business-like approach, presenting ideas logically and understandably, using tact and diplomacy, being more interested in a successful project than in limelight. After all, the client executive carries responsibility, and is entitled to any credit for results. A counseling relationship on the part of the consultant must be attained, which means the making of analyses and formulation of programs but not the making of decisions, and giving instructions to client people. These attributes are tools which the consultant uses. They are some of the points to bear in mind in selecting a consultant.

SUMMARY

The use of qualified management consulting firms to solve specific business problems has now become an acepted technique on the part of many progressive managements. The substantial growth in firms in the field attests to the need for consulting assistance for today's business executives.

When a client engages a consultant on a proper basis, he takes advantage of 1, an unbiased and objective viewpoint; 2, trained analysts; 3, a staff with specific experience; 4, uninterrupted application of time; and 5, the ability to present ideas and obtain action.

When he knows what to expect of a consultant and how to work with him, it usually pays off many times over.

Book Review

THE CHEMISTRY AND TECHNOLOGY OF FOOD AND FOOD PRODUCTS. Morris B. Jacobs, editor. Second revised and augmented edition. In three volumes, 2580 pages. Interscience Publishers, Inc., New York, N. Y., 1951. Price $42.00.

The first edition of this encyclopedic work appeared in two volumes in 1944. Reflecting the many new and important developments pertaining to foods, the second edition contains over 700 more pages. In contrast to the 48 chapters in the first edition, the present edition contains 52 chapters. The chapters have also been reshuffled into more logical groups. The three volumes contain 569 tables and 306 illustrations. Volume I now deals with the fundamentals of foods and food processes. This includes discussions of the physical chemistry of foods, the importance, occurrence and behavior of various chemical constituents as well as chapters dealing with digestion and food spoilage. Chapters on the principles of processing (unit operations, food machinery and control instruments, sanitation, quality control, etc.) complete this volume.

Volume II contains the chapters dealing with various foods (dairy products, meat products, poultry and eggs, confectionery, etc.), while volume III deals with the various types of food preservation processes and the technology of production. An excellent 77-page index embodying all three volumes completes this book.

The inherent faults of treaties dealing with broad fields rich in ramifications can be observed in this book. It is inevitable that some of the discussions are at times repetitions of discussions which may be found in other chapters. On the other hand, even with a certain extent of overlapping, not all corners of the subject are covered with the same thoroughness. To be sure, this second edition is much improved in such respects. Yet, short of having a genius devoting a number of years to the writing of a book covering such a tremendous subject like food, this is perhaps the best we can do with a complicated topic so interwoven with many other large and fast-moving fields like nutrition, engineering, biochemistry, etc. These discussions were mostly written by experts thoroughly familiar with the subject of a chapter while our enterprising genius could not possibly know his beans as well as his macaroni products. Therefore, we should be thankful for the courage and vision of the editor and his publishers, and to the forty-odd collaborating experts for producing this work.

To institutions and research laboratories in the food industries these volumes will serve as an easy means of obtaining summary information on almost any topic related to foods and food production. To smaller organizations who can not afford librarians and technical libraries, this work will well serve as a "library-in-one."

The typography is excellent and the binding and general appearance of the volumes are attractive.

Z. I. KERTESZ.

ANIMAL NUTRITION. By L. A. Maynard. 474 pages, McGraw-Hill Book Company, 1951. New York, N. Y. Third edition. Price $4.50.

This new third edition of Maynard's "Animal Nutrition" has been thoroughly revised, particularly the sections on intermediary metabolism, the vitamins, growth, and the use of isotopes in nutritional studies. It is 20 pages shorter than the second edition, but this reduction was accomplished by using narrower margins rather than reducing the material presented, and is well printed and physically well suited for textbook use.

The book is divided into four parts under the following headings, "General bases of nutrition," "The nutrients and their metabolism," "The measurement of body needs and feed values" and "Nutritive requirements for body processes and productive functions."

The author makes frequent reference to the classical studies of nutrition which tends to bridge the gap

between classical and modern nutritional theory. He draws upon the literature covering work with many species, including man, to illustrate the principles with which he deals. As Dr. Maynard points out, some of the fields are developing so rapidly that the student must consult the current literature, rather than a textbook. He has carefully indicated these areas where it is impossible to draw definite conclusions due to lack of information.

The book is most useful as a text for the study of the principles of animal nutrition. It gives the student with a background in the basic sciences an understanding of nutrition in those terms and makes available to him an introduction to the literature in the field. It is also of value to the research worker as a reference giving the overall viewpoint of nutrition.

The author is to be commended for keeping his book in the foremost position in its field.

F. H. KRATZER

SANITATION FOR THE FOOD-PRESERVATION INDUSTRIES. By the Association of Food Industry Sanitarians, Inc., in cooperation with the National Canners Association. 284 pages, McGraw-Hill Book Company, Inc., New York, N. Y., 1952. Price $5.00.

Adequately bound and printed on a well glazed paper, this book meets a decided need, particularly for the food processing industry, as it brings together a mass of heretofore disconnected information on the general topic of sanitation. This is the first compilation of its type available and is written in a style which is readable for the production manager as well as for those in charge of technical quality control.

The fourteen chapters include a variety of topics significant in quality control as it specifically relates to production and which may serve as a background for answering many of the problems which daily arise in connection with proper sanitary producers in food processing plants. Of particular merit are the chapters on maintenance and construction of building and equipment, the chapter dealing with detergents and cleaning compounds, and insect pests.

The discussion of cleaning and detergents is particularly well presented and should meet a need in giving valuable information on this subject to food plant operators. Although the information on the costs of cleaners assembled by those interested in formulating their own cleaning mixtures may become quickly obsolete, the methods of arriving at such costs will be of value. In this connection it might be indicated that the discussions relative to sanitizing are linked with chlorination and not with the many sterilizing procedures which might be used in sanitizing food processing equipment.

The physical cleaning of equipment is discussed with particular reference to design, a factor in quality control which has been too long overlooked. It is to be hoped that as sanitation is established as a unit process, equipment manufacturers will give more consideration to the design of equipment to facilitate ease of cleaning and sanitizing.

The chapter dealing with insects, pests, and their control is particularly noteworthy as information in this essential field of sanitation is generally not available to the average food processor.

The general field of sanitation insofar as food processing plants are concerned is well covered and particularly well documented with a bibliography cited in connection with each chapter. It is hoped that in future editions the sketchy outlines in certain sections will be enlarged. This book should be available to all production and quality control personnel.

G. J. HUCKER

CHEMISCHE KONSERVIERUNG VON LEBENSMITTELN. By Paul Hirsch. 150 pages. Theodor Steinkopff, Dresden und Leipzig, 1952. Price $1.68 (paper cover), $2.16 (bound).

This text on the chemical preservation of foods is number 54 of the *Technische Fortschrittsberichte* issued under the editorship of Dr. B. Rassow of Leipzig. It is divided into 10 sections rather than chapters. The principal topics covered are chemical preservatives, the action of chemical preservation, testing the suitability of preservatives both on a laboratory and industrial scale, the application of chemical preservation (including blanching), German legal specifications, use of the oligodynamic effect, and a group of topics treated briefly covering antibiotics as preservatives, gas storage, coating, potato storage, preservation with ammonia, egg preservation, and packaging. Salting, pickling, smoking, acidification, sugaring, use of fats and oils, and antioxidants are also briefly discussed.

The chemical, pharmacological, and antimicrobial properties of formic acid, benzoic acid, sodium benzoate, *p*-chlorobenzoic acid, *p*-hydroxybenzoic acid and its esters, hexamethylenetetramine, boric acid, sulfur dioxide (sulfurous acid), and hydrogen peroxide (all of which are permitted by the law of the German Democratic Republic (East Germany), April 22, 1949) and of salicylic acid, cinnamic acid, formaldehyde, hydroquinone, hydrogen fluoride, and some of lesser significance such as thiourea, 8-hydroxyquinoline sulfate are discussed. Methods for the assay of these preservatives are detailed. The laws of the German Democratic Republic governing the use and labeling of preservatives are quoted and the amounts permitted are tabulated.

In the section dealing with the oligodynamic effect, its use (principally silver) in water sterilization is described and methods for the determination of silver are detailed. An interesting point mentioned is the use of streptomycin for the preservation of human milk.

The introductory section on foodstuffs concerning proteins and amino acids, carbohydrates, lipids, vitamins, and minerals is not up-to-date and could very well have been omitted. A few minor errors were noted such as misspellings of the names of American authors. The book is relatively well printed and bound and it has both subject and author indexes.

This book has interesting information but since virtually all of the chemical preservatives mentioned (with the exception of the benzoates) are banned in this country, its usefulness is limited to those interested in the general topic of chemicals in food products and in what chemicals and practices are permitted legally in a foreign country.

MORRIS B. JACOBS